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PATENT APPLICATION
Docket No. 6300.96.1

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re application of

A. Bruno Frazier et al.

Serial No.: 09/787,498

Filed: March 16, 2001

Conf. No.: 3953

For: SURFACE MICROMACHINED MICRONEEDLES

Confirmation No.: 3953

Examiner: Kathryn Odland

) Art Unit
) 3743

DECLARATION UNDER 37 C.F.R. § 1.131

We, A. Bruno Frazier and John D. Brazzle declare as follows:

1. We are the coinventors of the invention claimed in the above-identified patent application Serial No. 09/787,498. We were previously employed by The University of Utah, in Salt Lake City, Utah.

2. During our employment at The University of Utah and prior to June 10, 1998, we conceived the idea of surface micromachined microneedle devices as described and claimed in the above-identified patent application in this country. Copies of invention disclosure documents describing and showing various inventive concepts of the presently claimed invention and their reduction to practice are attached hereto as Exhibits A and B.

3. All of the claimed embodiments of the invention were conceived prior to June 10, 1998, while some of these embodiments were reduced to practice prior to June 10, 1998, and others were reduced to practice with due diligence after June 10, 1998.

4. Exhibit A comprises laboratory notebook pages 1-42, which include written descriptions, drawings, manufacturing procedures, and photographs of various microneedle array embodiments of the invention. Each of the dates deleted from Exhibit A are prior to June 10, 1998.

5. Photomicrographs of microneedle array devices of the invention fabricated prior to June 10, 1998 are shown on pages 28, and 35-37 of Exhibit A.

6. Exhibit B comprises laboratory notebook pages 43-81, which include written descriptions, drawings, manufacturing procedures, and photographs of single microneedle embodiments of the invention.

7. Photomicrographs of single microneedle devices of the invention fabricated after June 10, 1998 are shown on pages 50, 51, 65, 66, 68, 69, 80, and 81 of Exhibit B.

8. We declare that all statements made herein of our own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful, false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful, false statements may jeopardize the validity of the application or any patent issuing thereon.

Dated this 1st day of April 2004.

Inventor:


A. Bruno Frazier

Dated this 1st day of April 2004.

Inventor:


John D. Brazzle

John D. Boff

Needles

Frazier 355-3076

1

Meeting w/ Frazier @ 1500 hrs on 16 Apr 97.
- possible project on a 3-Dimensional Micromachined
Needle Array. Would be in charge of
Company Requirements

Phase I: Demonstration that a two-dimensional microneedle array and manifold can actually be made.

The dimensions, etc. of the array and manifold for this phase would be: (if you think there are better dimensions, indicate where you would select different dimensions)

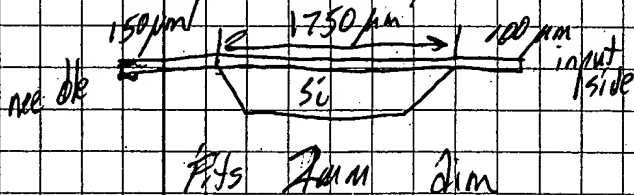
- overall array size 5mm x 5mm square
- needle spacing 200u centers--the array would be 25x25
- needle size 75u wide, less than 75u thick on the outside 40u wide by greater than 10u on the inside
- needle length 150u from base of needle to tip of needle
- needle tip geometry 75 deg bevel on narrow dim, 45 deg on wide dim
- manifold thickness less than 3 mm
- volume of manifold 1mm x 5mm x 5mm (on the inside) (above needle array and below the luer)
- syringe interface standard luer

Deliverables: 10 devices of the above geometry, greater than 90% of needles are open per array, when placed on a syringe fluid is expressed from the manifold with roughly equal velocity (and the manifold is capable of handling the internal pressures--less than 100 PSI--without breaking).

From
~~XXXXXXXXXX~~
~~XXXXXXXXXX~~
Anonymous
CO.

Start Date: ~ June or sooner (awaiting contract approval)
End Date: phase I for one year
personal: attempt to finish by Christmas

Conceptual Design Ideas:



Thickness Consideration:

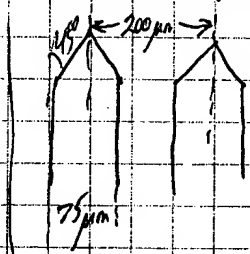
25 needles thick: w/ max 75 μm structure
need 125 μm thick Si wafer

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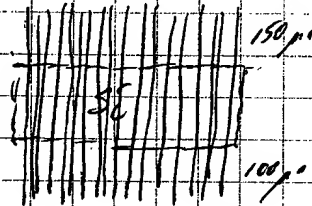
Could design structure to be around 60 μm high, like Si wafer
e.g. 6 mils = 150 μm
slightly beyond 5 mm requirement
OK

John D. Byrd

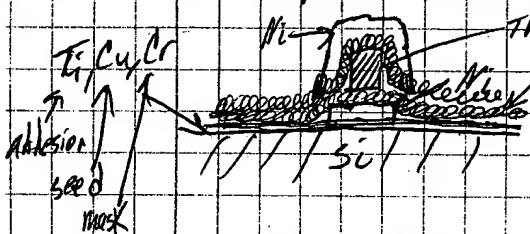
25 Needles Wide



leaves $62.5 \mu m$ et ss on either side of array



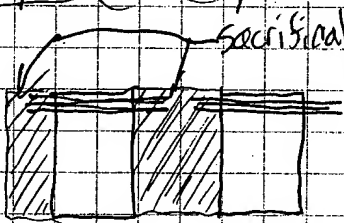
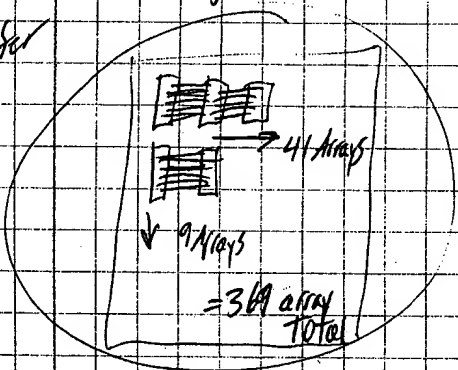
will utilize existing PR procedure to form hollow rectangular channels



Apply PR, etch Cr, plate Ni
spatter Au, Apply thick PR, pattern
spatter Au, pattern PR, plate Ni
strip Thick PR in Acetone bath

Water layout

2" water
N-type



sacrificial boron membranes
use phosphorous doped substrate.

= 14 devices (25x23) 100%
= 11 75%
= 7 50%
= 3 25% yield on water

Use L-edit for Mask design.

Need: KOH Mask -1
Bottom shell -2
Thick PR -3
Top shell -4

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Cool idea!

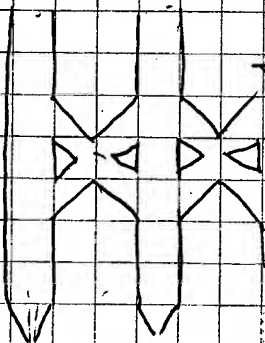
[Signature] 3

90% Needles open per array
roughly equal velocity in each needle is equal pressure.

— from T-jet port injection manifold use in
later model cars.

50% ideal

V-engine



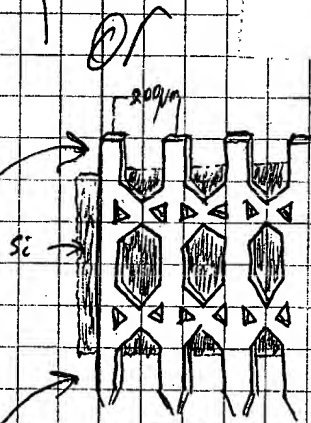
→ 25 Needle array

Discussed with Frazer

[Signature]

Mainly Nickel structure

Ta or Ti inside channels for
bio compatibility.



structures
built over
sacrificial membranes

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4
Phil

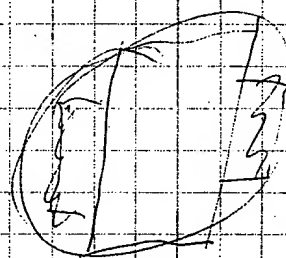
John & Beryl

French use of Pd in place of Ni for structure.

Contact: Mr. abys @ Lucent Technologies,
Bell Labs, 908-592-6408

John & Beryl

Pd cool literature Pd



Hardness: 430-500 KHN₂₅

purity: 99+ Pd

Density: 11.7 g/cm³

Weight: 2.9 mg/cm² for 2.5 micron

John & Beryl

possible present palladium to group as replacement of e-plating Ni and Au.

John & Beryl

Think about mask dimensions

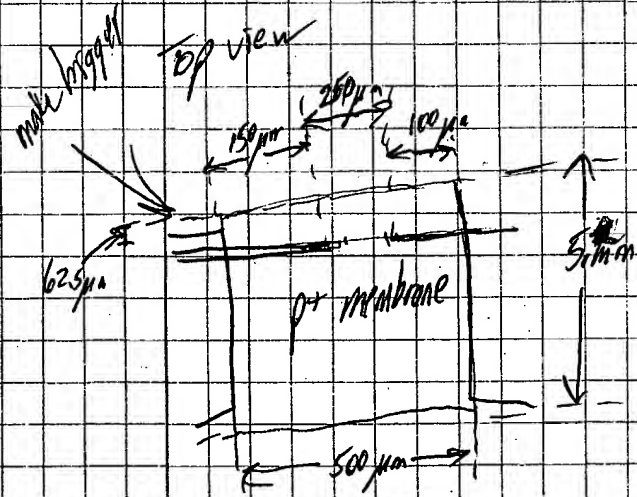
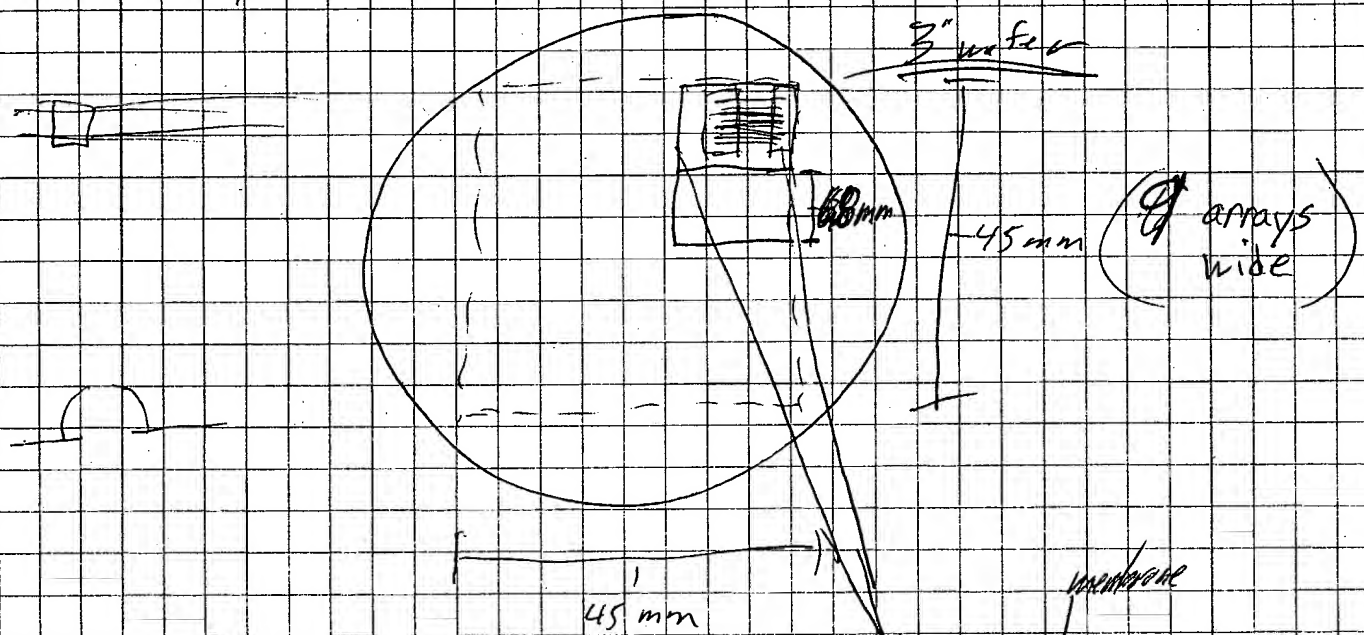
Mask Design - 3 masks total

bottom shell
same as
top shell

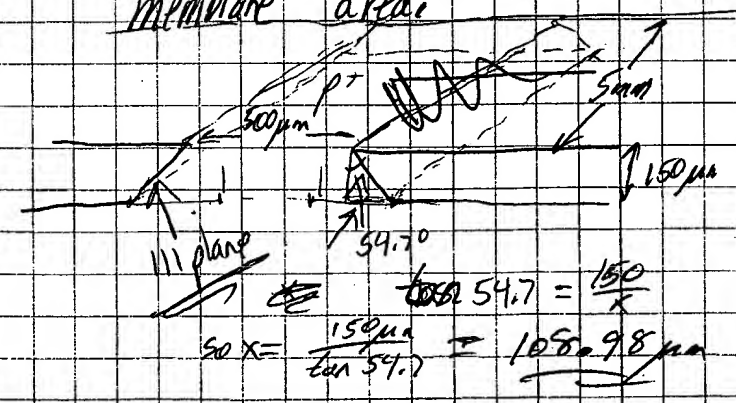
Mask #1 LOH mask

- to define sacrificial membrane and alignment marks

- using 45 mm² area on a 3 inch wafer

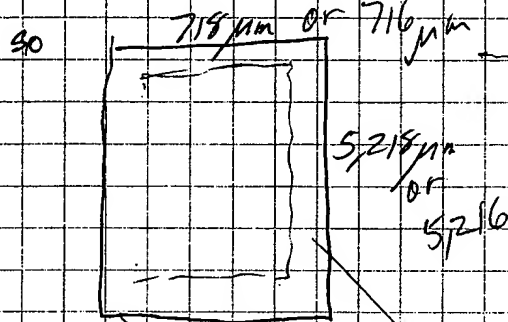


membrane area:

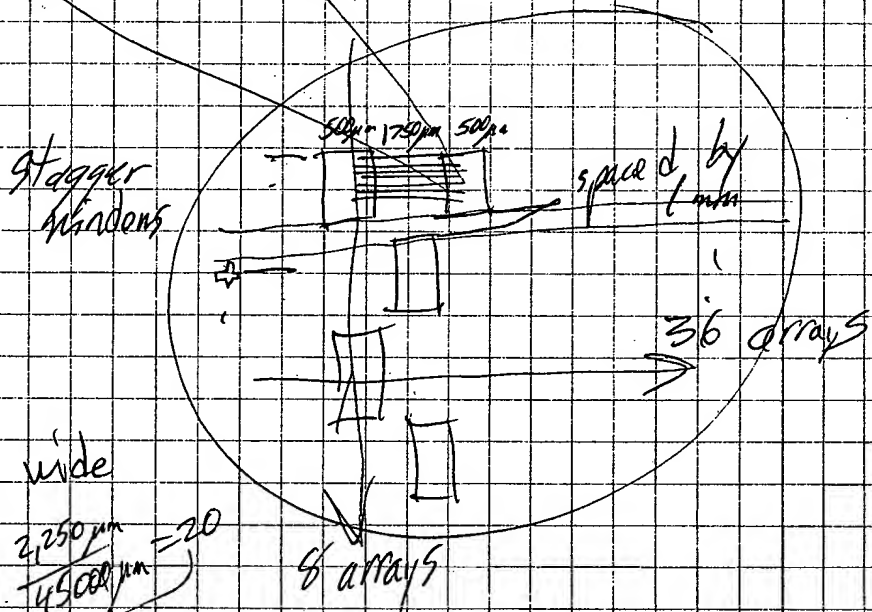


Mask #1 continued:

So window dimensions for mask:
 are: width: $500\mu\text{m} + 2x = 500 + 2(109) = 718\mu\text{m}$
 and length: $5\text{mm} + 2x = 5\text{mm} + 2(109) = 5.218\text{mm}$



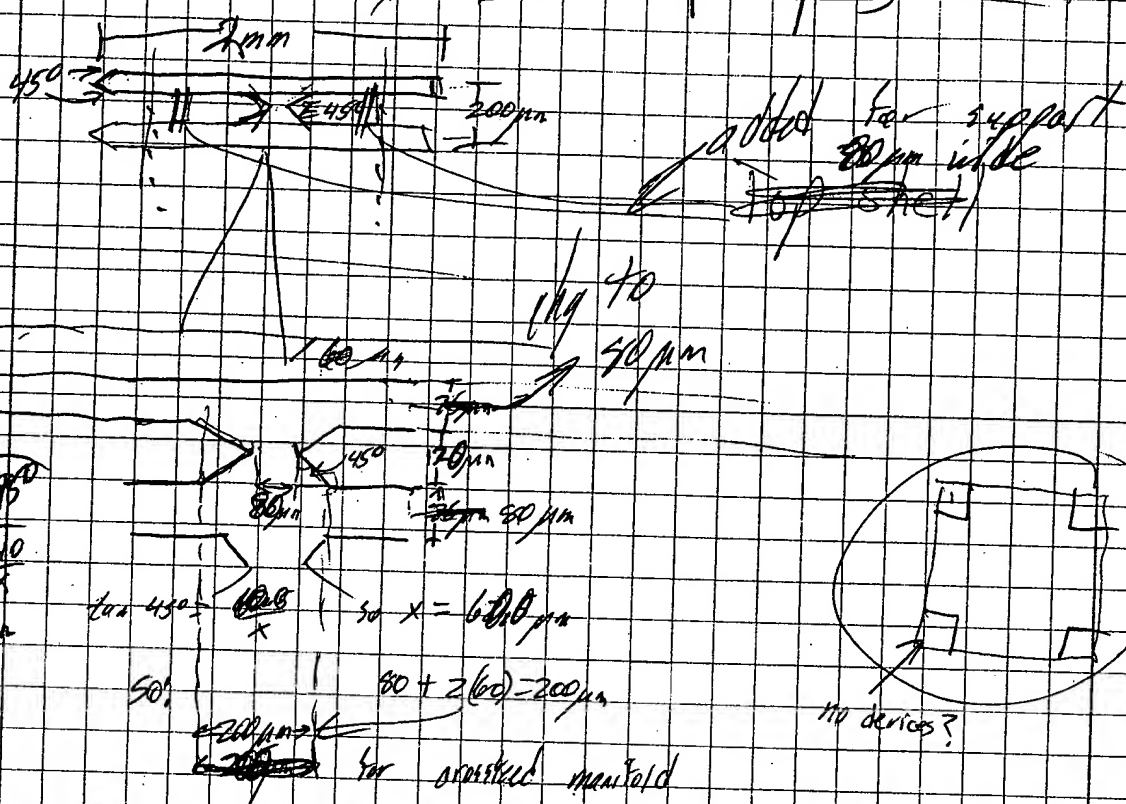
or 108 μm
 taking into consideration
 10% etch in
 planar
 111 μm
 1.5 μm



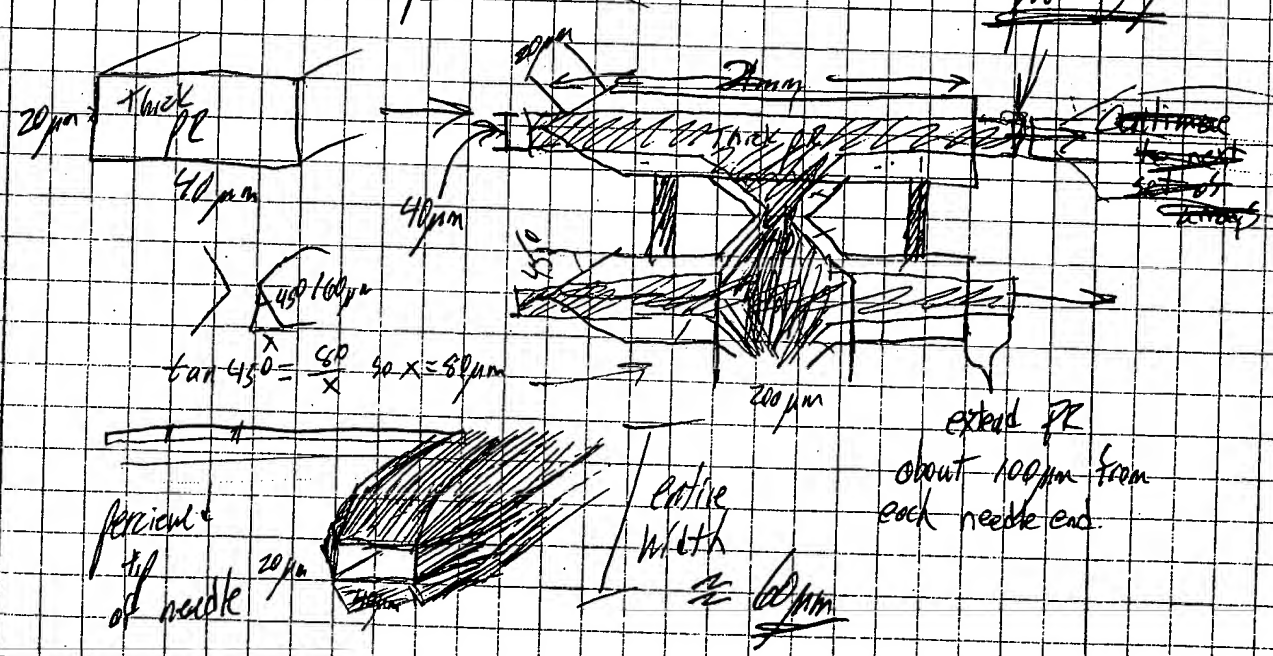
so 8 arrays wide
 by 36 long
 $\frac{2,250\mu\text{m}}{45,000\mu\text{m}} = 20$

Change
 to 2mm
 length of needle

Mask #2 Bottom & top etching mask



Mask #3 Thick PR mask

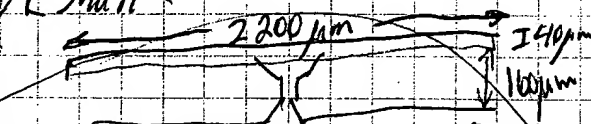


Mike

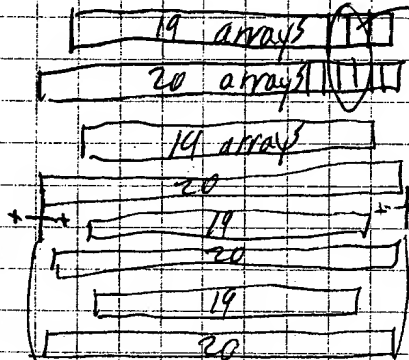
performing mask designs on 'Z-edA'

or Mask

array width: 4350 μm

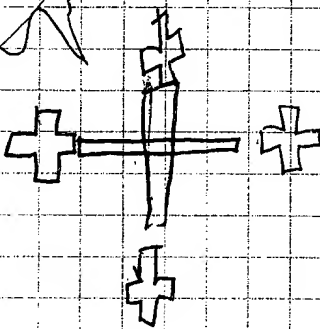


off center staggering for member stability



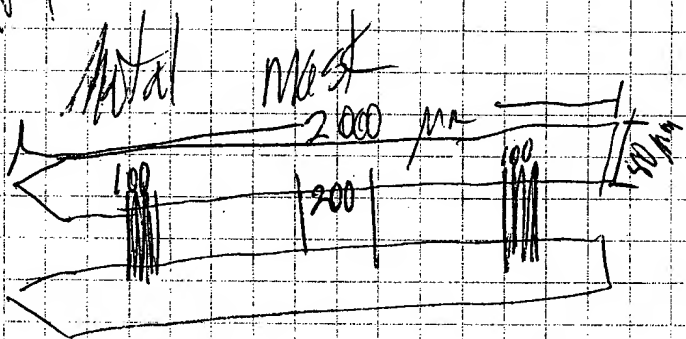
= 156 total arrays

alignment marks



Translation: 4350 μm

array width: 4350 μm



Q

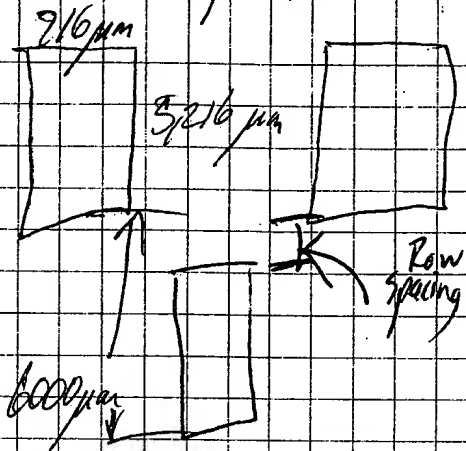
LOH mask

Dimensions:

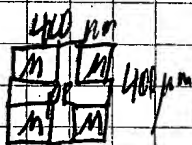
spacing, $1750 - 2(108) = 1534 \mu m$

widths 5,216

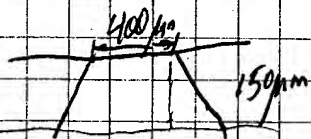
Translation:
X: -354
Y: -108
(for mask alignment)



alignment marks:



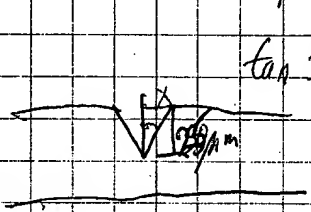
X: -24,75
Y: 24,141
X: 24,54
Y: 14,42



$\tan 54.7 = \frac{150}{x} \Rightarrow x = 109.98 \mu m$
Dims are $400 + 2(108) = 616 \mu m$
so need $616 \mu m^2$ alignment marks

Scribe marks:

want to etch $\approx 36 \mu m$ into substrate



$\tan 54.7 = \frac{15}{x} \Rightarrow x = 35.7 \mu m$

so make LOH lines 35 μm wide

so:

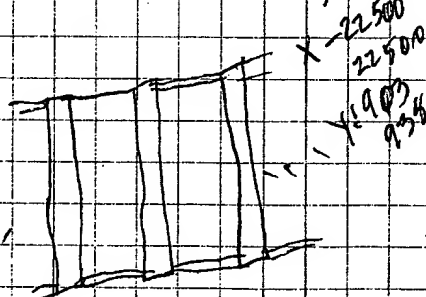
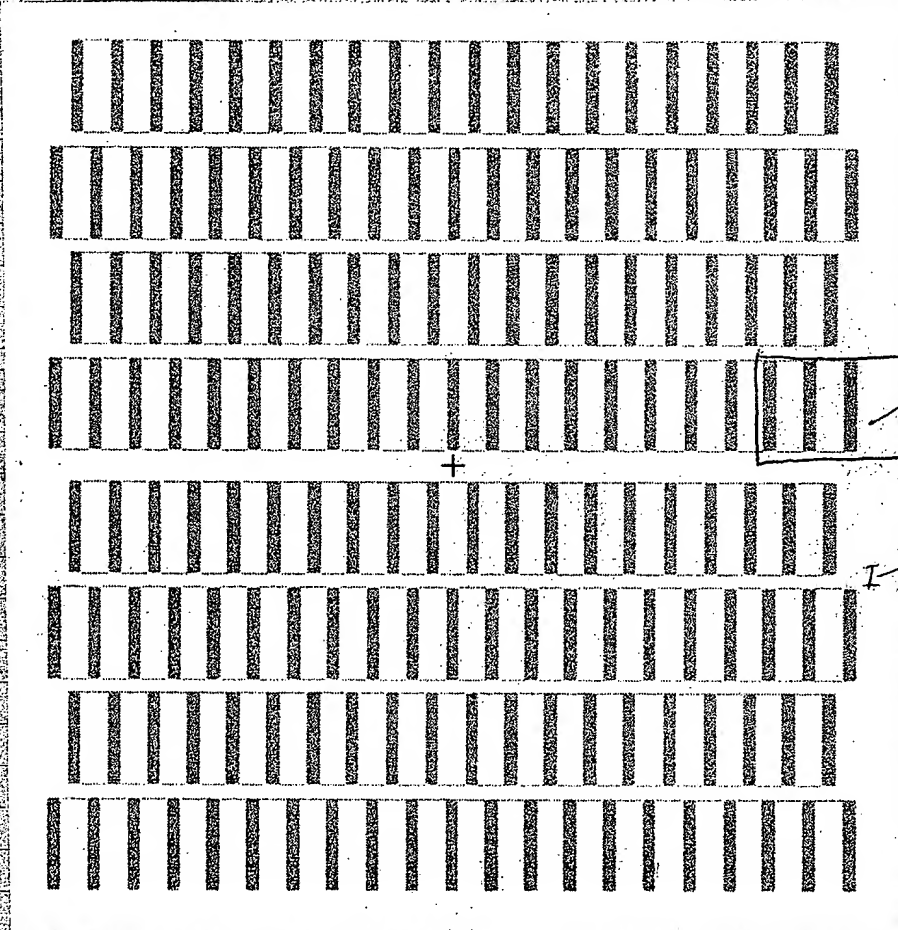




Photo Dept

Completed Mask Designs in '2 edit'
ROH Mask

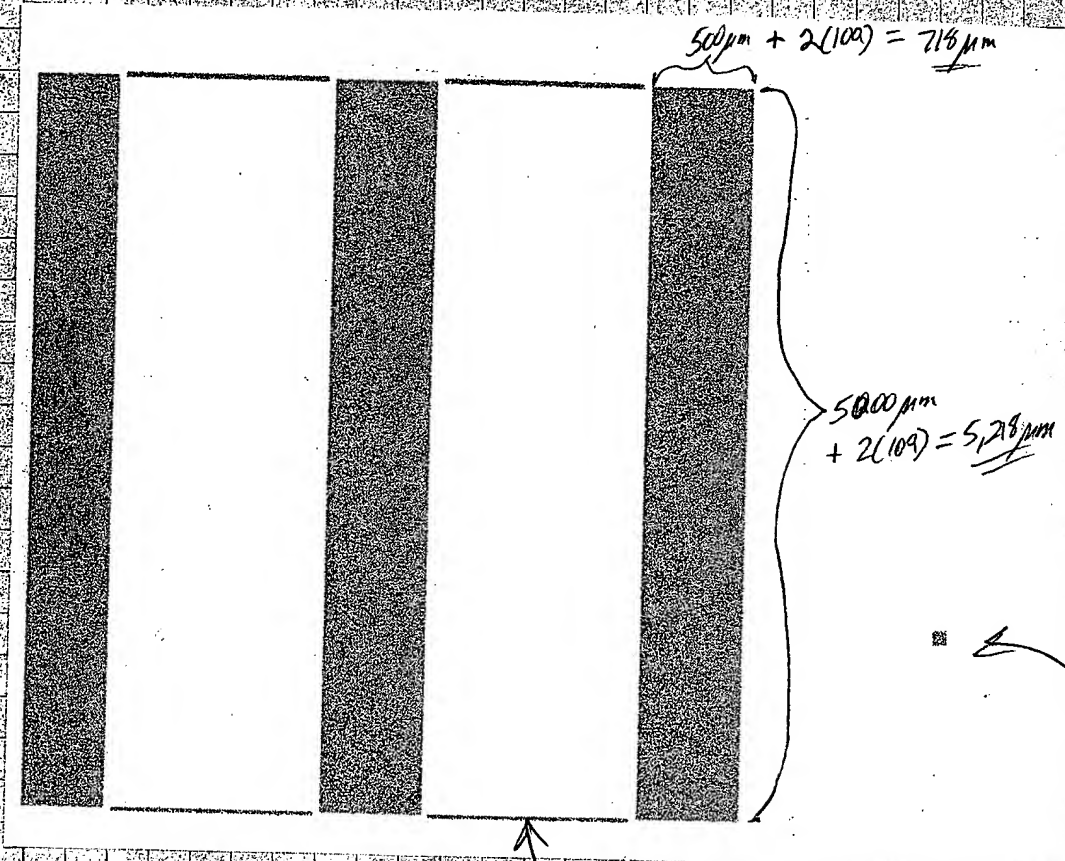


Blown up

I am

Photo B

Abel



$$500 \mu\text{m} + 2(100) = 716 \mu\text{m}$$

$$5000 \mu\text{m} + 2(100) = 5,200 \mu\text{m}$$

Scribe lines
35 μm width

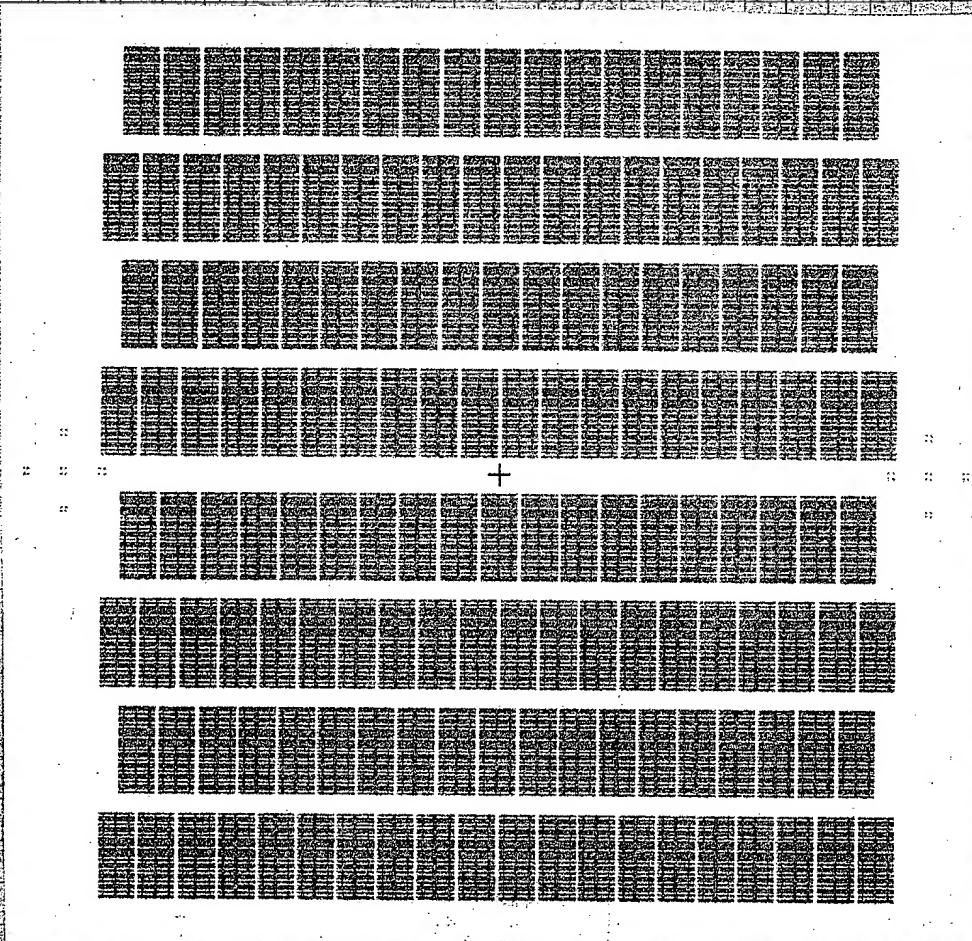
Alignment Mark
100 μm

12

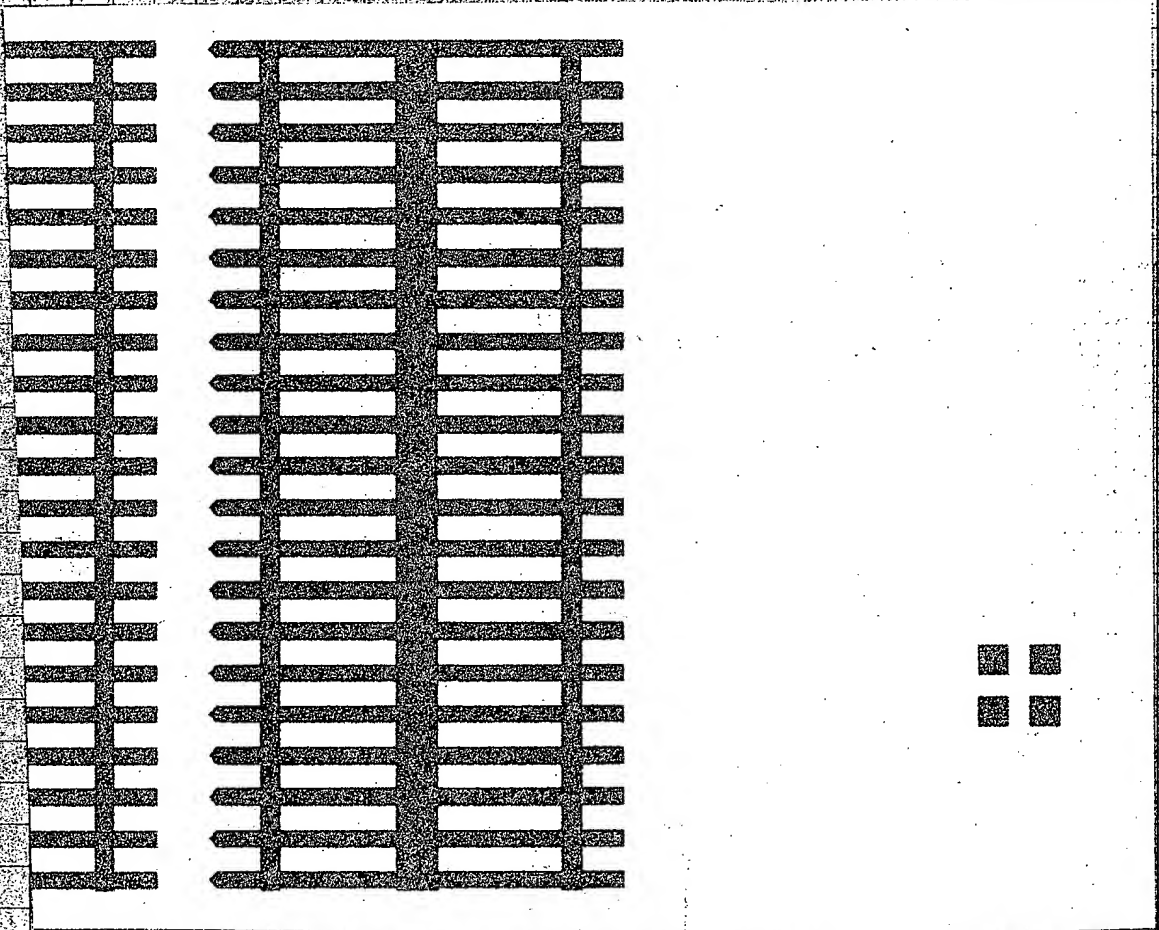
Metal

Mask

12



[Handwritten signature]



Pha

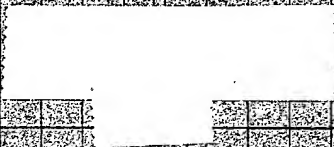
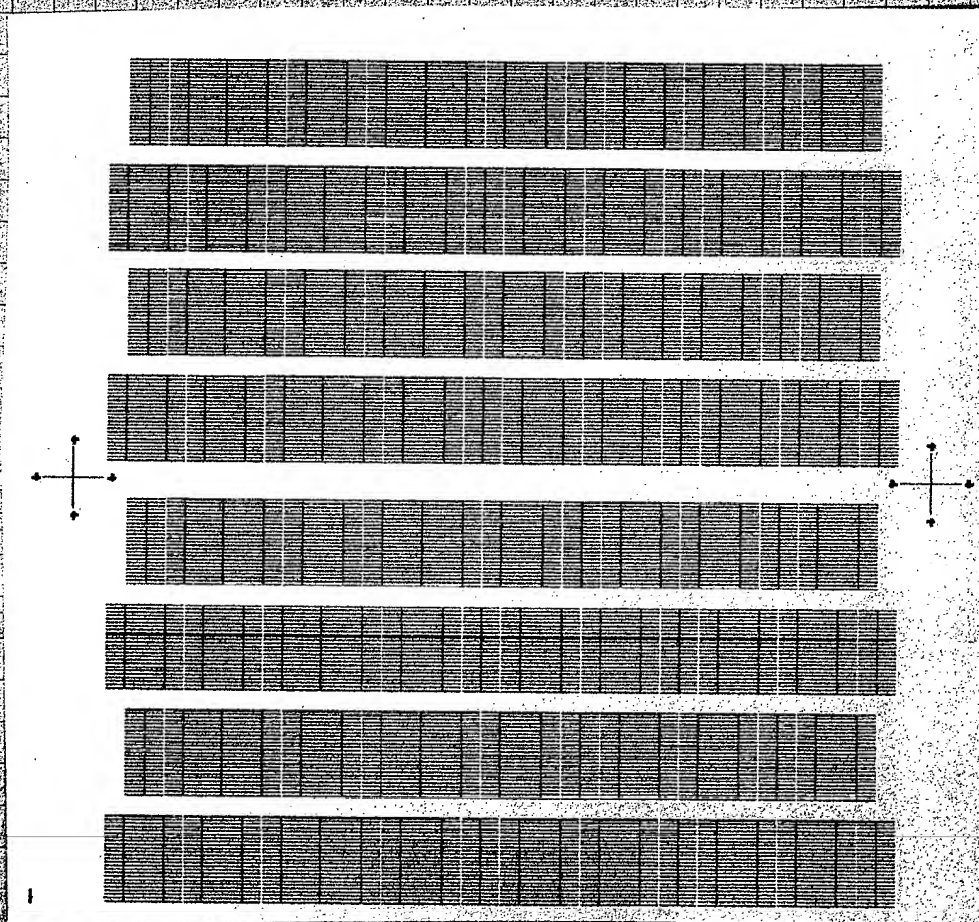


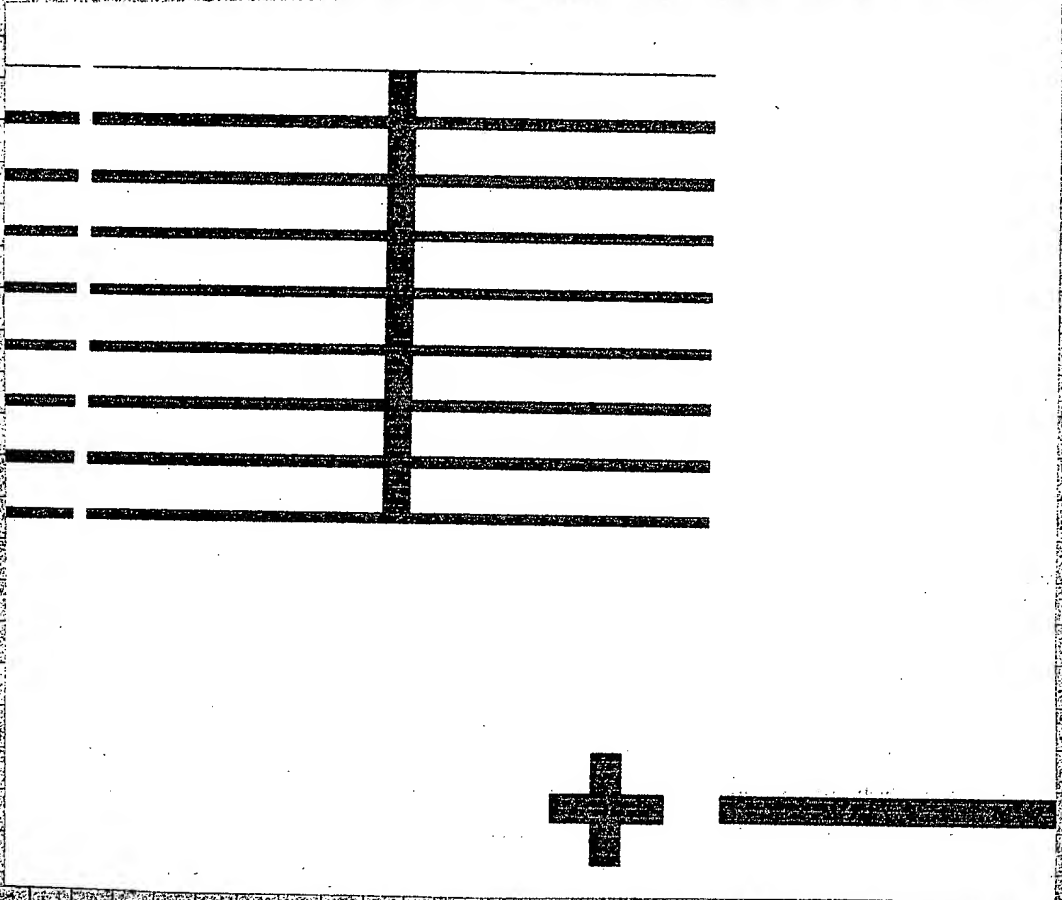
photo resist

Mask

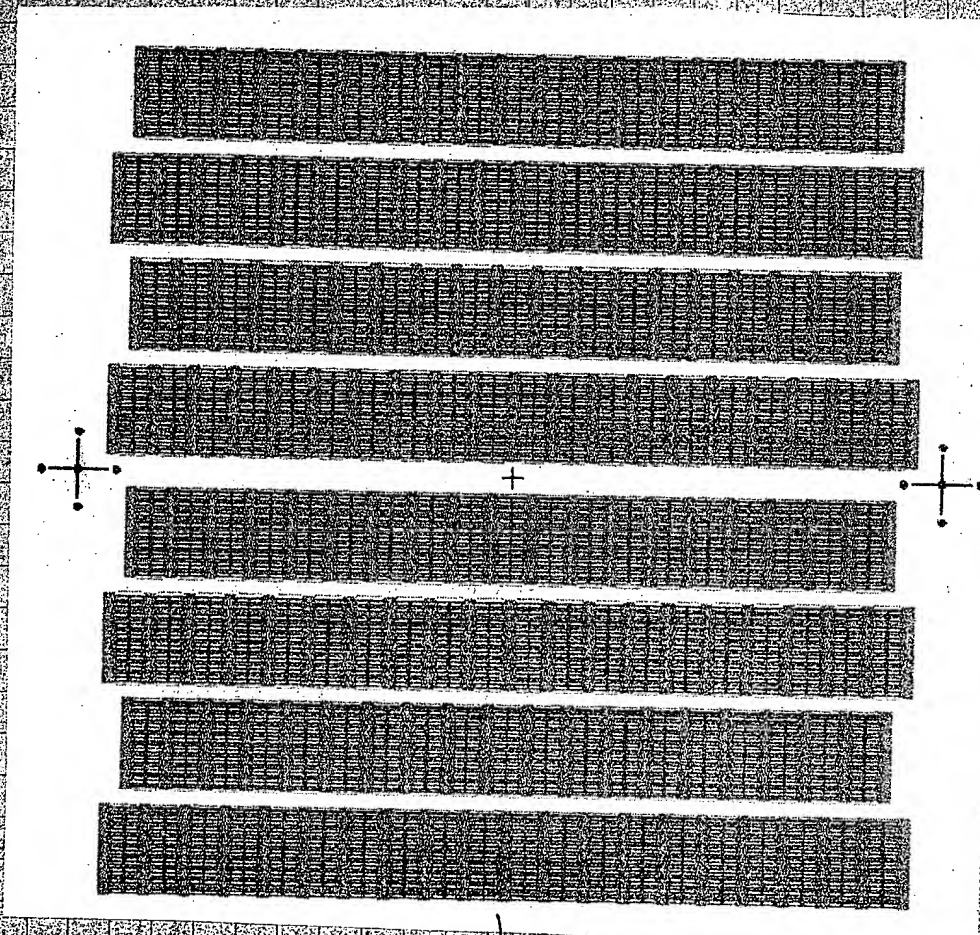
Hand



[Handwritten signature]

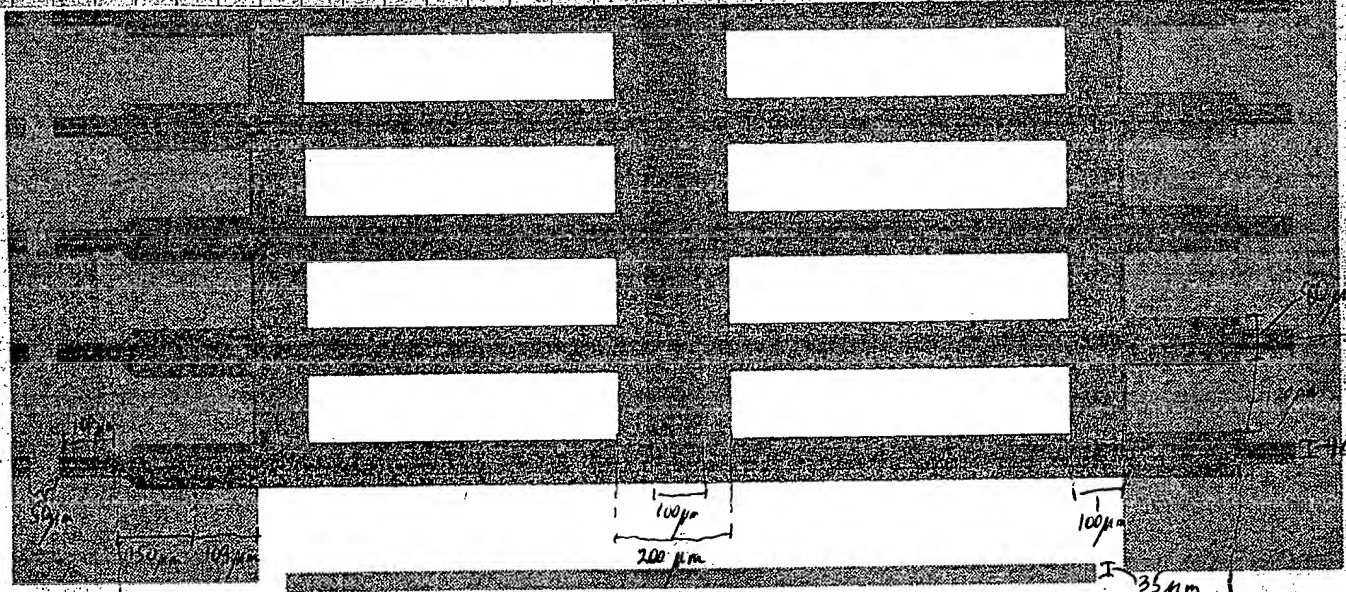
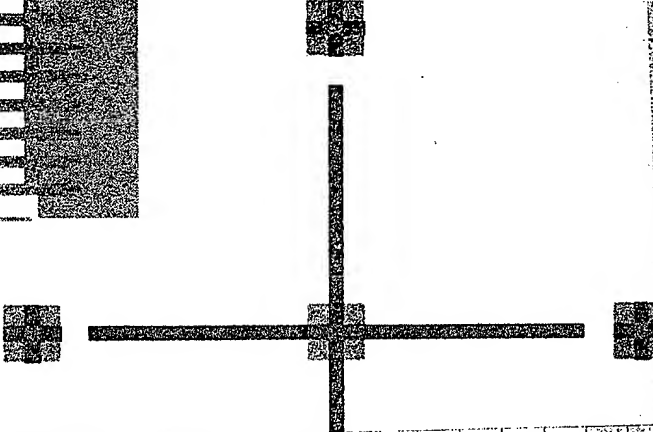
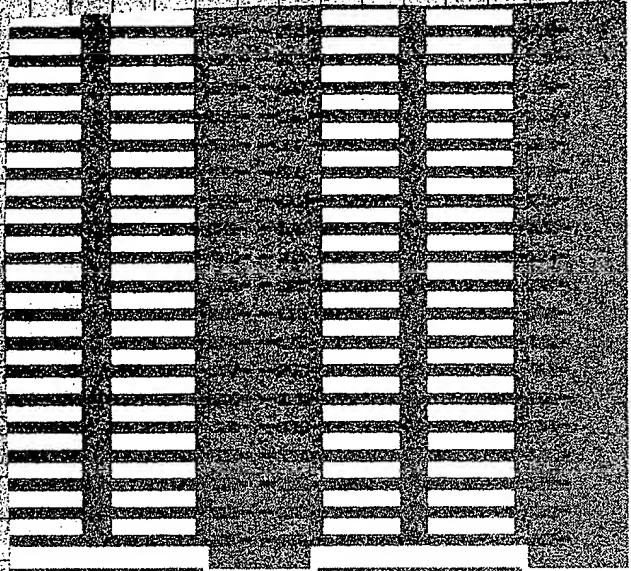


All Tree Mass



[Handwritten signature]

17
[Handwritten signature]



50 μ m

50 μ m

100 μ m
200 μ m

100 μ m

35 μ m

50 μ m

50 μ m

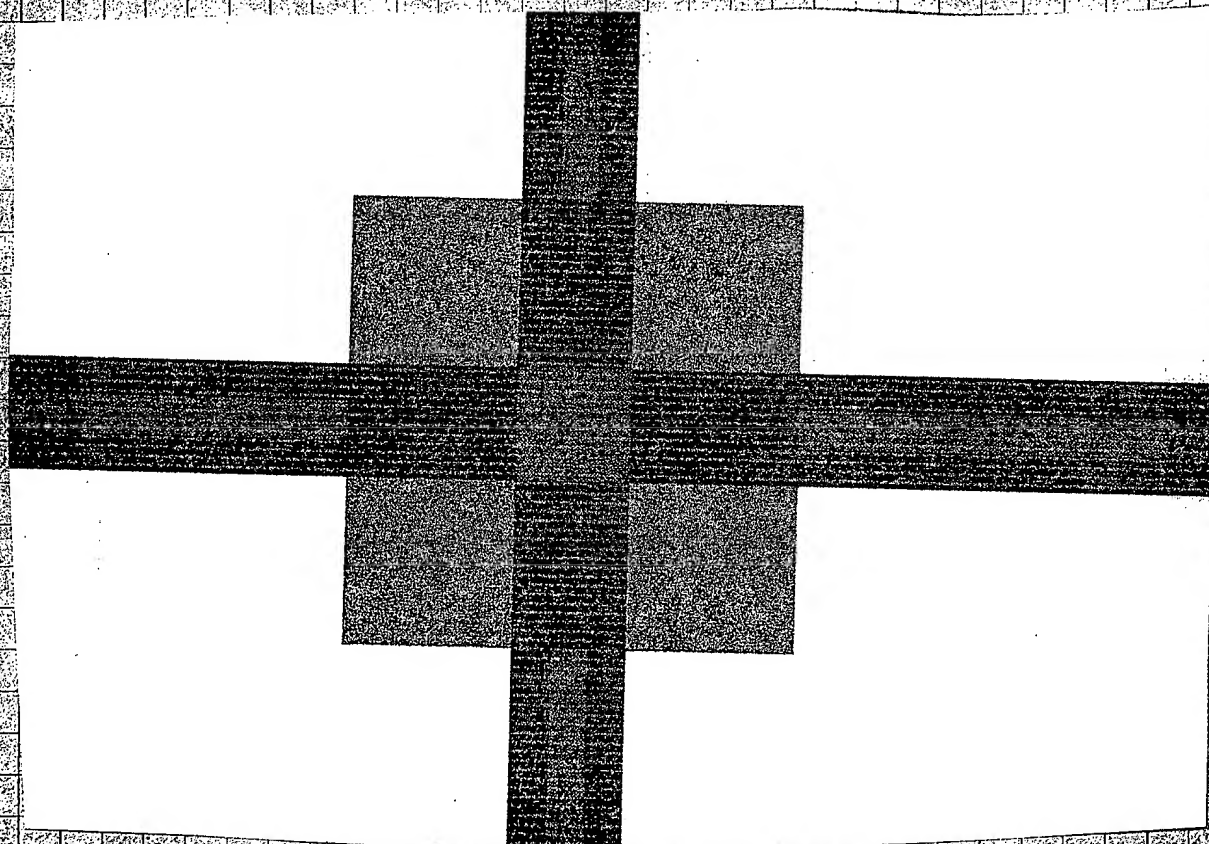
50 μ m

2 mm

A
H
A

Jh. 0. 1/2

Alignment Marks (Center)



Masks in Fabrication

19

1- Water preparation (pt doping)

- Modified RCA clean

4 min piranha-etch 70 ml H_2SO_4 / 30 ml H_2O_2

- rinse DI 2 min

30 sec HF (5%)

- rinse DI 2 min

~~4 min Metal-etch 33 ml H_2L / 33 ml H_2O_2 / 33 ml H_2O~~

4 min Metal-etch

33 ml H_2L / 33 ml H_2O_2 / 33 ml H_2O

- rinse DI 2 min

30 sec HF (5%)

- rinse DI 5 min

* check wafers for hydrophobic character

~~4 min Metal-etch~~ Boron Sources aging

800°C

30 steel O_2

95 steel N_2

5 hours

- Boron Diffusion (in 3mm)

1175°C

11 glass O_2

131 steel N_2

5 hours

H&H

John D. [Signature]

3- prep for Si_3N_4 deposition

- HF dip (50%) 3 min

- DI Rinse 5 min

+ Si_3N_4 deposition using PECVDSubstrate temp: 300°C N_2 flow: 15 sccm NH_3 : 55 sccm SiH_4 : 23 sccm

RF power: 40 W - 2 sec pulsed

HF power: 4 W - 4 sec pulsed

pressure: 300 m Torr

Time: 45 min each side of wafer

4- ~~KOH~~ etching (anisotropic)

- pattern:

PR 1813

1500 rpm

30 sec

1000 rpm

40 sec

expose: 15 sec

develop: AZ 352

= 30 sec

- RIE Nitride etching

CF₄ flow

40% (40 sccm)

power

50% (150 W)

pressure

300 m Torr

Time

≈ 8 min

- water clean



Acetone 15 sec
Methanol 15 sec
piranha-clean 2 min
DI Rinse

70% Sulfuric 30% H₂O₂

- Kott Bath

30g Kott to 1200 ml H₂O

Temp: 60°C

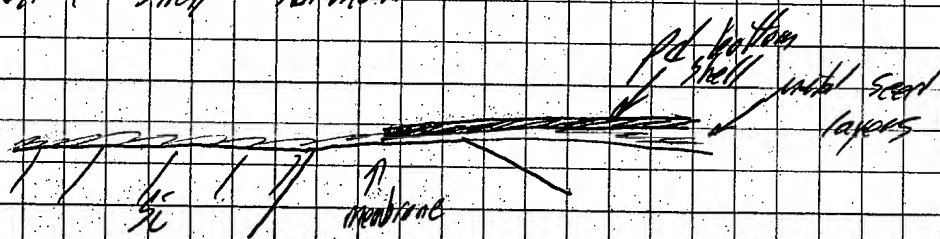
Time: ~ 7.5 hours

Note: slow agitation of bath mixture w/ plastic mesh to break-up films

- DI Rinse 5 minutes
N₂ blow dry (low pressure)

4 May 97

5- Pottery shell formation



- Metal seed layers (sputter)

Power: 250 W
Area: 60 cm²
Vacuum: 1×10^{-6}

Times:

Ti → 4 min ≈ 600 Å
Cu → 2 min ≈ 1500 Å
Cr → 3 min ≈ 1000 Å

→ go to pg 25
for process data

Thick PR process development

1st attempt: (on gold covered wafers)

PR 4620

300 rpm

15 sec

800 rpm

20 sec

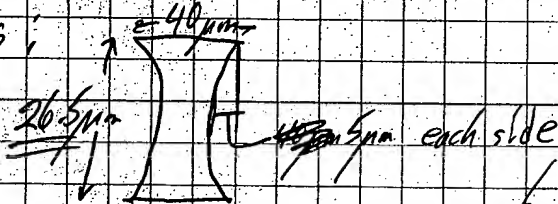
Cure: 110°C

2 min

Expose: one bar 20 sec
" " " 30 sec

develop: 40 ml 400K : 100 ml H₂O
± 5-8 min

Results:



30 sec exposure

looks better!

(if possible reflection problem)

2nd attempt

Make Anti-Reflective Coating
ARL

1500 rpm 20 sec
2000 rpm 60 sec

Cure: 130°C 20 s
145°C 1 min

PR 4620

300 rpm

15 sec

950 rpm

20 sec

Cure 110°C

2 min

Expose: 40 sec, 30 sec, 20, 10

develop: 400K
Bad Results

2nd attempt

used

50 s 40 s 30 s 20 s

No APL

40 s looks best

4th attempt

40 sec looks perfect.

procedure:

PR 4620

300 rpm

15 sec

950 rpm

20 sec

Cure

110°C

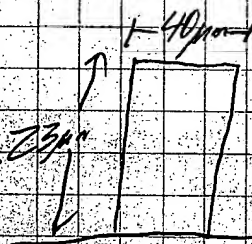
2 min

Expose

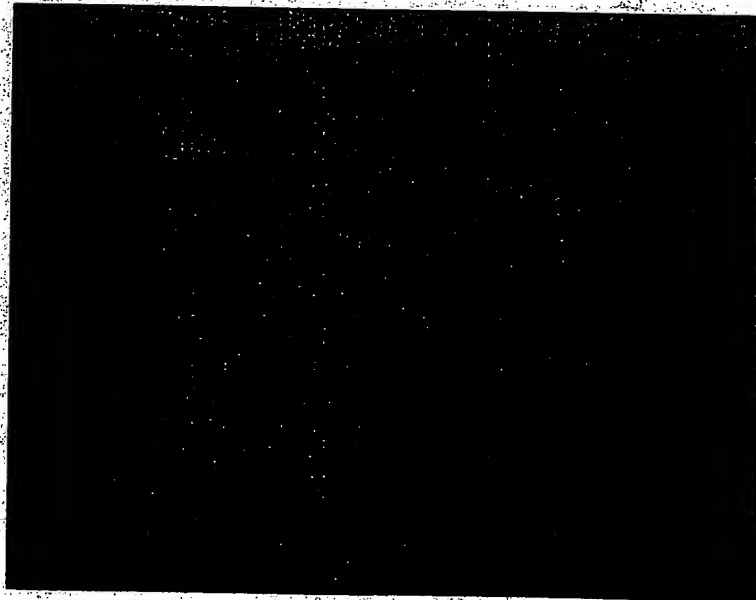
40 sec

Develop

400K

~~2.5:1~~40 ml 400K : 100 ml H₂O

actually →



Sauter on thick PR

Attempts: 1- 10 min @ 100W RF $\approx 1200^\circ$

Failed - left bubbles in PR

2- 5 min @ 100W RF $\approx 600^\circ$

Failed -

3- 2.5 min @ 100W RF $\approx 300^\circ$

GOOD!

Need $\approx 1000^\circ$

So:

Use
this

4- 2.5 min @ 100W RF

then 15 min cool

2.5 min @ 100W RF

then 15 min cool

the 2.5 min @ 100W RF

yields $\approx 1000^\circ$ GOOD appearance

Thick PR over encapsulated PR

4620 speed 300 rpm 20 sec \rightarrow GOOD

1000 rpm 20 sec

Make 370C (oven) 25 min \rightarrow GOOD

Expose 40 sec \rightarrow Bad

Rinse 400C

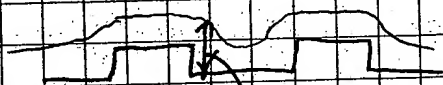
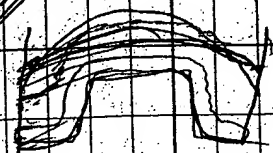
2.75:1 30 ml 400C

Did Not Work \rightarrow Next try 50 s + 60 s



suspected underexposure

John D. Rupp



this region is thicker than 23μ
make $27-35\mu$ so increase

exposure next time.

problems with water sticking to mask,
need to cure longer

- Trial @ 35 min Cure time
results: GOOD cure

exposed for 55 sec NOT GOOD enough
need to increase

John D. Rupp

All Dummy waters trashed!

Have 2 waters w/ TiCl₄/Cr

* pattern for bottom shell *

→ PE 4620 300 rpm 15 sec

1000 rpm 20 sec

Bake 110°C 2:15

expose 45 sec

Develop 35 ml 400K / 100 ml H₂O

results: GOOD side wall appearance!

- E-plating of bottom shell

Cr-etch 75% HCl / 25% H₂O

Pd plating 30°C questionable bath?

Current Density: 5 mA/cm² Area = 3.037 cm² Total

pd plating cont.

$$5 \text{ ma/cm}^2 \times 8.04 \text{ cm}^2 = 40.2 \text{ mA current setting}$$

plating Time: 1 hr \approx 17 μm

Results: Adhesion problem

possible ~~thin~~ stresses

tensile (peel-up)

$$\text{at } 3.73 \text{ ma/cm}^2 \times 8.04 \text{ cm}^2 = 30 \text{ mA current setting}$$

plating Time: 45 min

~~Bad Adhesion~~

Bottom shell w/ 1 hour nickel and 10 min Pd
w/ membranes, add 1000 rpm, 30 sec dry PR, 3 min @ 1000

fasten Thick PR: exposure at 50 sec was better

thick PR on bottom shell:

PR 4620 300 rpm 15 sec
1000 rpm 20 sec

bake 110°C 2:15 min

expose 55 sec (on membrane water)

develop: 35 ml 400°C / 100 ml H₂O

Note: Exposed for 45 & 30 sec on surface waters
30 looks a little better

Sputter:

100 w RF Au for 2.5 min

cool 15 min

100 w RF Au

for 2.5 min

cool 15 min

100 w RF Au

for 2.5 min

Note: 50 sec exposure looks best

Top shell Micro mold

John D. Bly

PT 4620 300 rpm 20 sec
1000 rpm 20 sec

Bake 37°C (oven) 35 min

Expose: 60 sec

Develop 35 ml 400K / 100 ml H₂O

Stack to mask
Increase Cure Time

John D. Bly

Top shell Cure time @ 60 min @ 37°C

GOOD!

Expose: 60 sec

Develop 400K

GOOD!

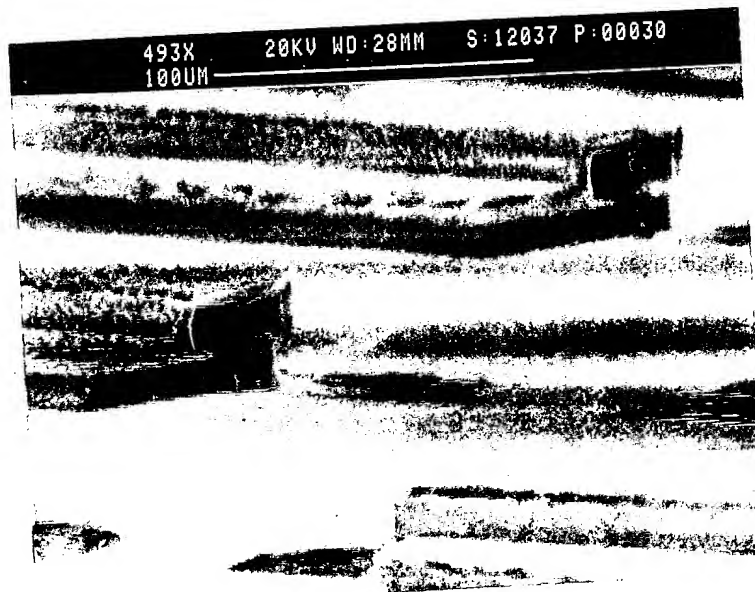
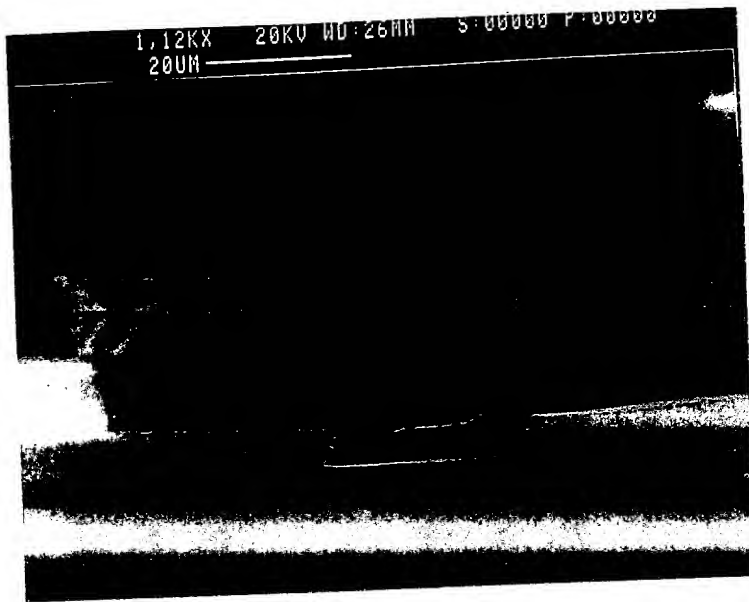
John D. Bly

Top shell etching

10 min per @ 5 mA/cm²

1 hour Nickel @ 10 mA/cm²

SEM of surface work



Notes: Arriving later waters
from Silicon Quest. Export arrived on 11 Dec.

~~John D. Long~~
If waters in bath are washed!
Si₃N₄ lifted completely off the
unpolished side.

14 Dec 92 ~~John D. Long~~
Took two waters and did 1.75 hrs
of Si₃N₄ on the unpolished side and 45 min
on polished side.

~~John D. Long~~ Bath @ 10°C start time 1 am. 20 Dec
300 g KOH / 1200 ml H₂O

Results: after only 3 1/2 hours, the KOH
was etching the entire surface due to
roughness of the unpolished side.

~~John D. Long~~ Alternate Technique: Using one of the existing
waters w/ completed surface work.

Lift-off procedure

etch Chromium: 50% HCL \approx 2 min

etch Copper: Ammonium Hydroxide saturated
with Cupric sulphate. \approx 1.5 hours

Backside of wafers too rough
 Try SiO₂ as mask for KOT
 Need to characterize a 3" furnace!

Furnace failed several characterization attempts. Furnace found to be malfunctioning.
 Parts on order

3" Furnace still inoperative. attempting to fine up new 4" Furnaces - awaiting electricians starting ANSYS Finite element modeling of the needles.

Met w/ Finzier.. decided current wafers will not work. Double-sided polished flexible wafers on order. Also will use lift-off technique to see if assembly is possible.

List of procedure for fabrication -

started w/ 6, 10-12 ml thick, new waters
performed standard water clean.

Deposit Metal seed layer system (sputtered)

Power: 250 W
Argon: 60%
Vacuum: 1.2×10^{-6}
Pressure: 4.77
Flow: 55.7

Times + Metals

Ti \rightarrow 4 min \sim 600 Å
Cu \rightarrow 3 min \sim 2250 Å
Cr \rightarrow 3 min \sim 1000 Å

Pattern Bottom Shell

PR 4620

300 rpm
1000 rpm

15 sec
20 sec

Rate NOPL

2:15

expose:

45 sec

Develop

40% 400X/100 ml 450

Bottom shell e-forming

etch Cr in 75% HCl/25% H₂O

pl bath: pH: 8.1

Current density: 5 mA/cm²

Current Setting: 5 mA/cm² \times 8.04 cm² = 40.2 mA

plate time: 1 hr yields: Adhesion Problem

Adhesion problem investigation

- found pin-holes in Cr layer,
etch didn't remove all Cr before
plating. possible mono layer of Si_3N_4
on the Chromium.

prepared new batch of wafers

- clean
- Si_3N_4 both sides
- Cu , Cr
- patterned 2 wafers w/ bottom shell

wafer #

plate @ $40.2 \text{ mA} = 5 \text{ mA/cm}^2$

Time: 1 hr

Thickness: $\approx 5 \mu\text{m}$

Results: Good Adhesion

Water #2:

plate pd @ 80 mA = 10 μ m

1 hr \approx 10 μ m

Good adhesion but very rough!

patterned 2 more wafers w/ bottom shell

Water #1:

Current: 40.2 mA

Time: 3 hours

Thickness: \approx 17 μ m

Results: poor adhesion (possible PR problem)

same w/ #2 water

Must solve pd adhesion problem

Bottom shell formed w/ nickel

1 hr @ 120 mA (15 mA/cm²) yielded \approx 18 μ m

thick PR on bottom shell

300 rpm 15 sec, 1000 rpm 20 sec, bake 110°C 2-15 min
expose 50 sec, dev. 40 ml 100% room temp H₂O

~~AK~~
 7/20/78

4th

100 W RF 2.5 min

cool 15 min

100 W RF 2.5 min

cool 15 min

100 W RF 2.5 min

~~Top shell micro mold~~

prepared two more wafers of above

Top shell micro mold

pr 4620

300 rpm 15 sec

1000 rpm 20 sec

Cure in oven @ 37°C for 60 min

Expose 60 sec

Develop 100m H₂O / 40m 400K

Top shell etching

1 hr metal @ 120 mA (15 mA/cm²)

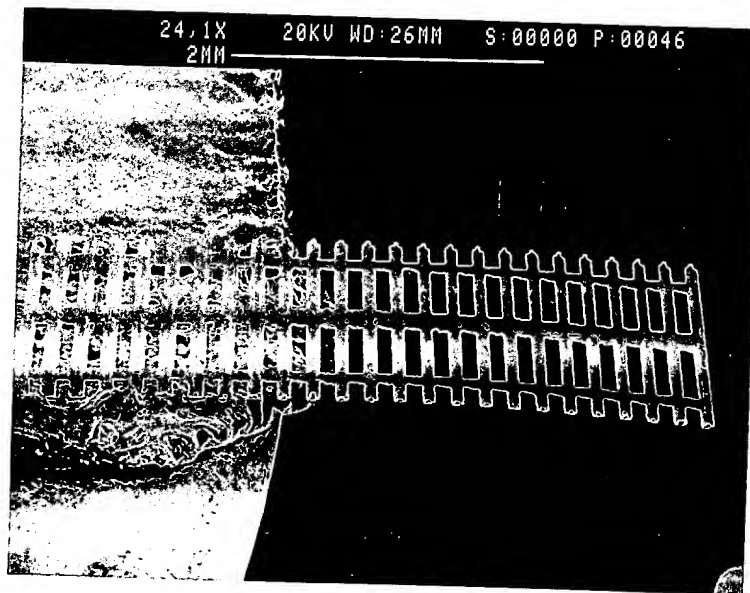
Results: Good!

John D. B. [signature]
 Lift-off procedure (Time between each step)

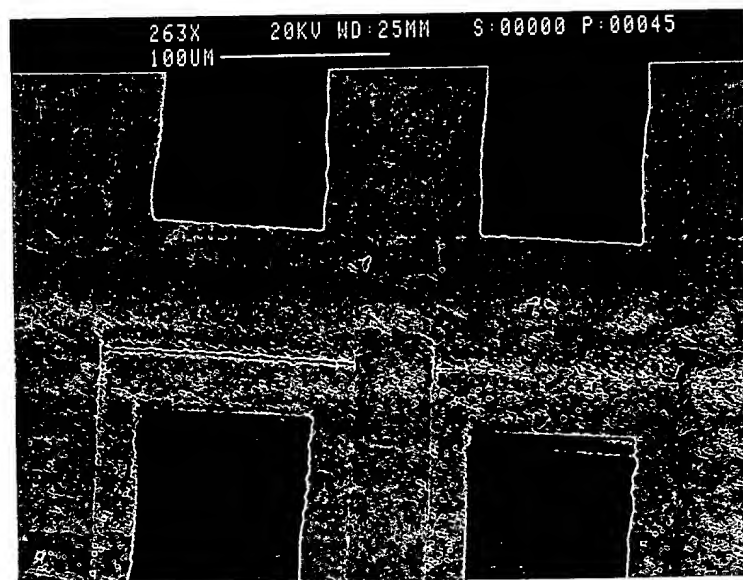
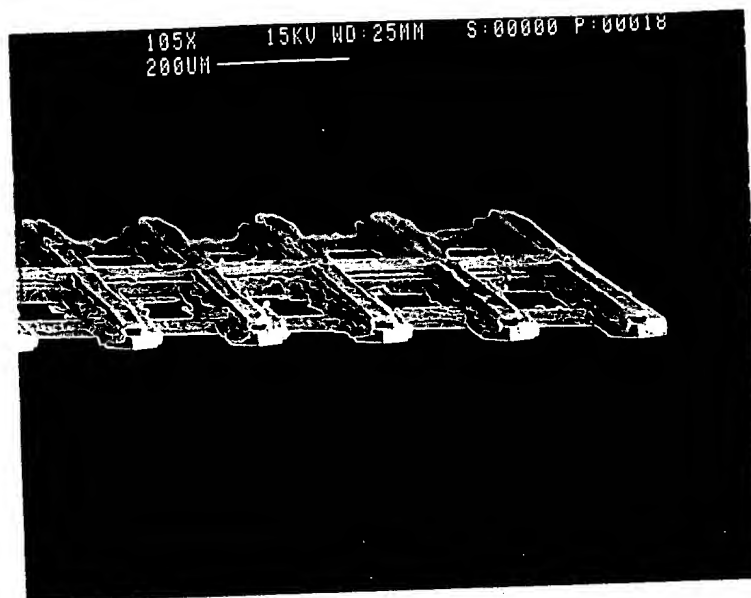
- 1- 30 sec "Gold etch"
- 2- 5 min Acetone
- 3- 2 min 2-propanol
- 4- Cr etch in 75% HCL \approx 2 min
- 5- Copper etch to lift-off \approx 1.5 hours

(Cupric Sulphate saturated in Ammonium Hydroxide)

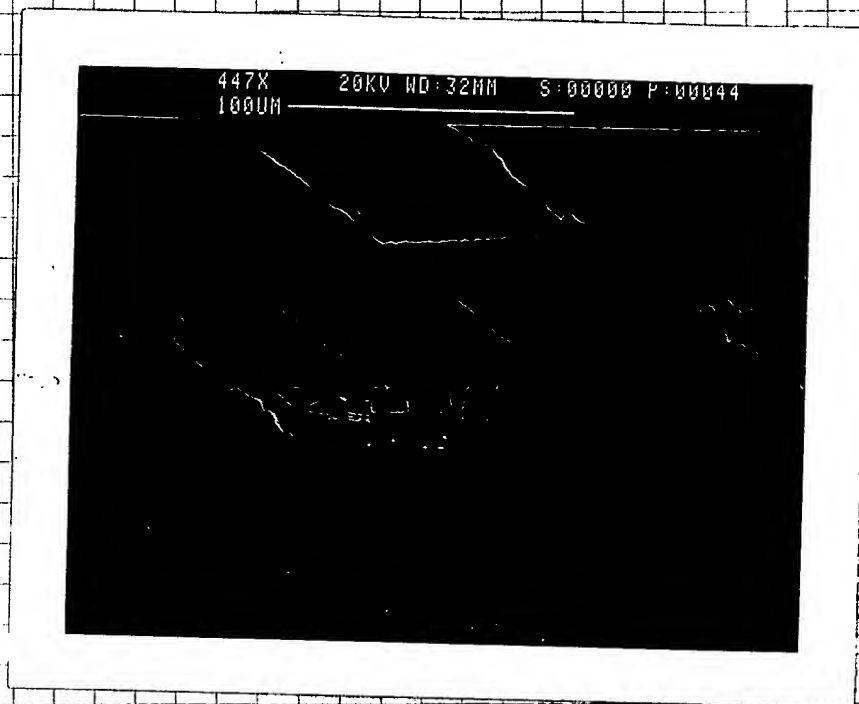
preparation for SEM



Array of 25 needles

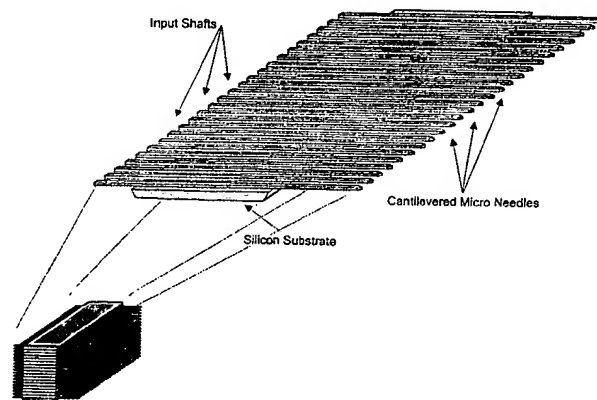


Needle Coupling Channels



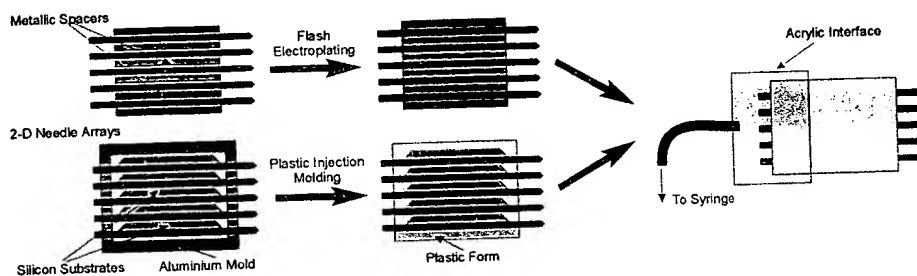
$\approx 20 \times 30 \mu\text{m}^2$ id

Consider Methods for assembly of 2D
devices into a 3D device



possible methods for assembly into
a 3-D device:

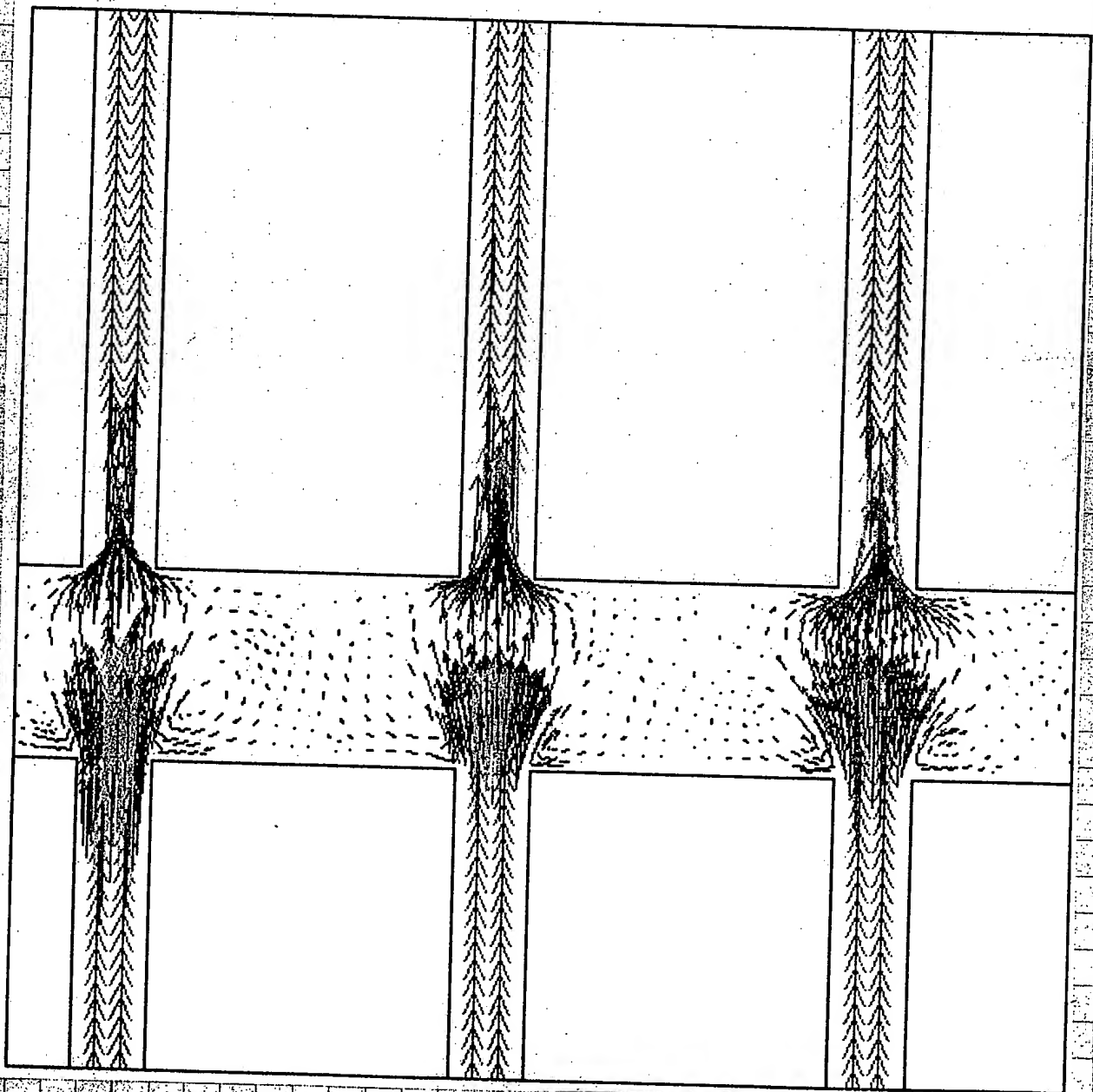
- 1- Use 150 μ m thick, double-sided tape.
- 2- Flash e-plating w/ released devices and metallic spacers.
- 3- plastic injection molding w/ aluminum mold.



*ANSYS Modeling to determine
fluid flow through fluid coupling channels*

ANSYS 5.4
APR 8 1998
14:17:31
VECTOR
STEP=1
SUB =1
V
NODE=9829
MIN=0
MAX=1.471

0
.183822
.367645
.551467
.735289
.919111
1.103
1.287
1.471



10 day

ANSYS 5.4
APR 8 1998
13:39:02

VECTOR

STEP=1

SUB =1

V

NODE=8660

MIN=0

MAX=1.661

0

.207641

.415283

.622924

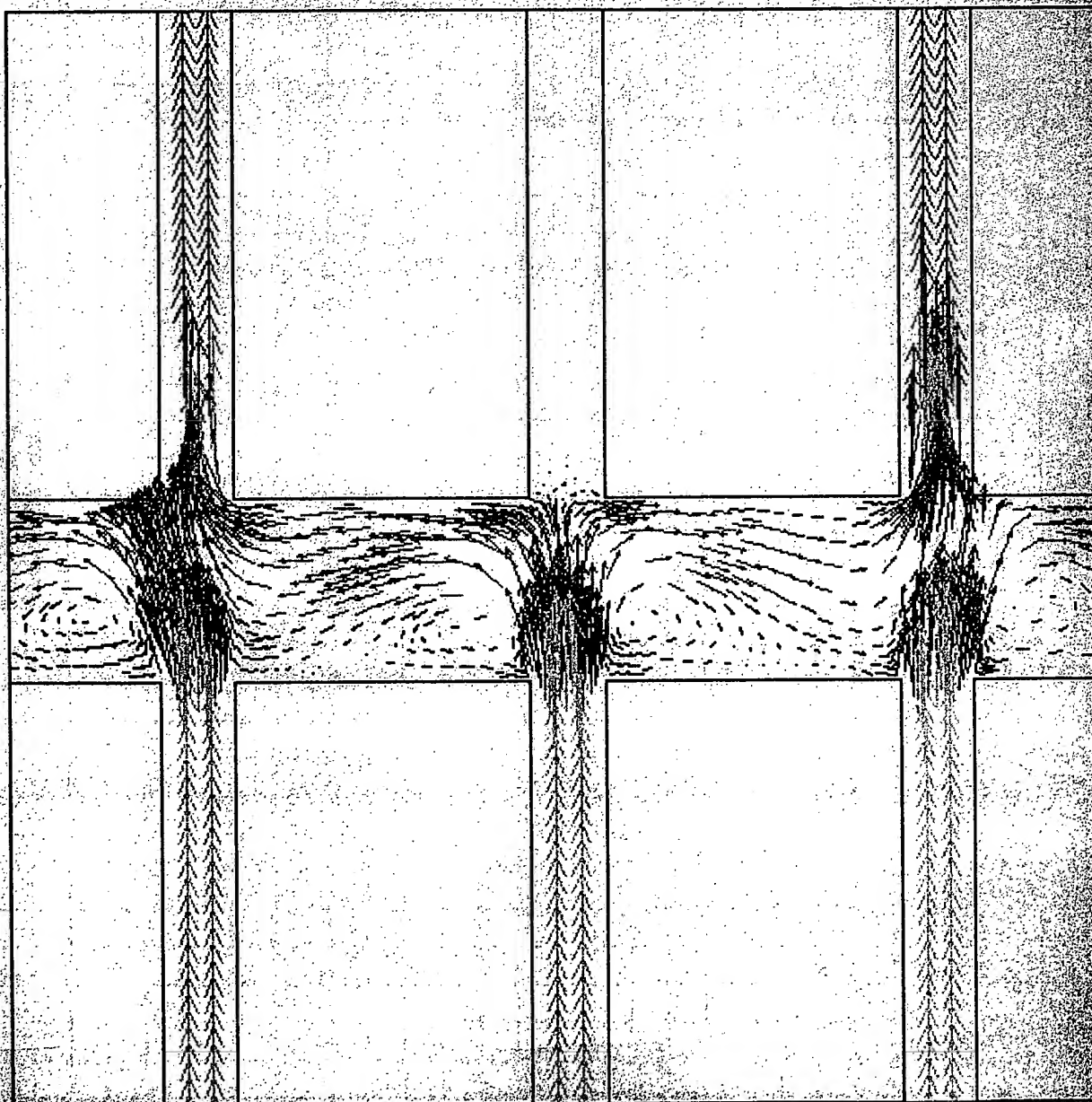
.830565

1.038

1.246

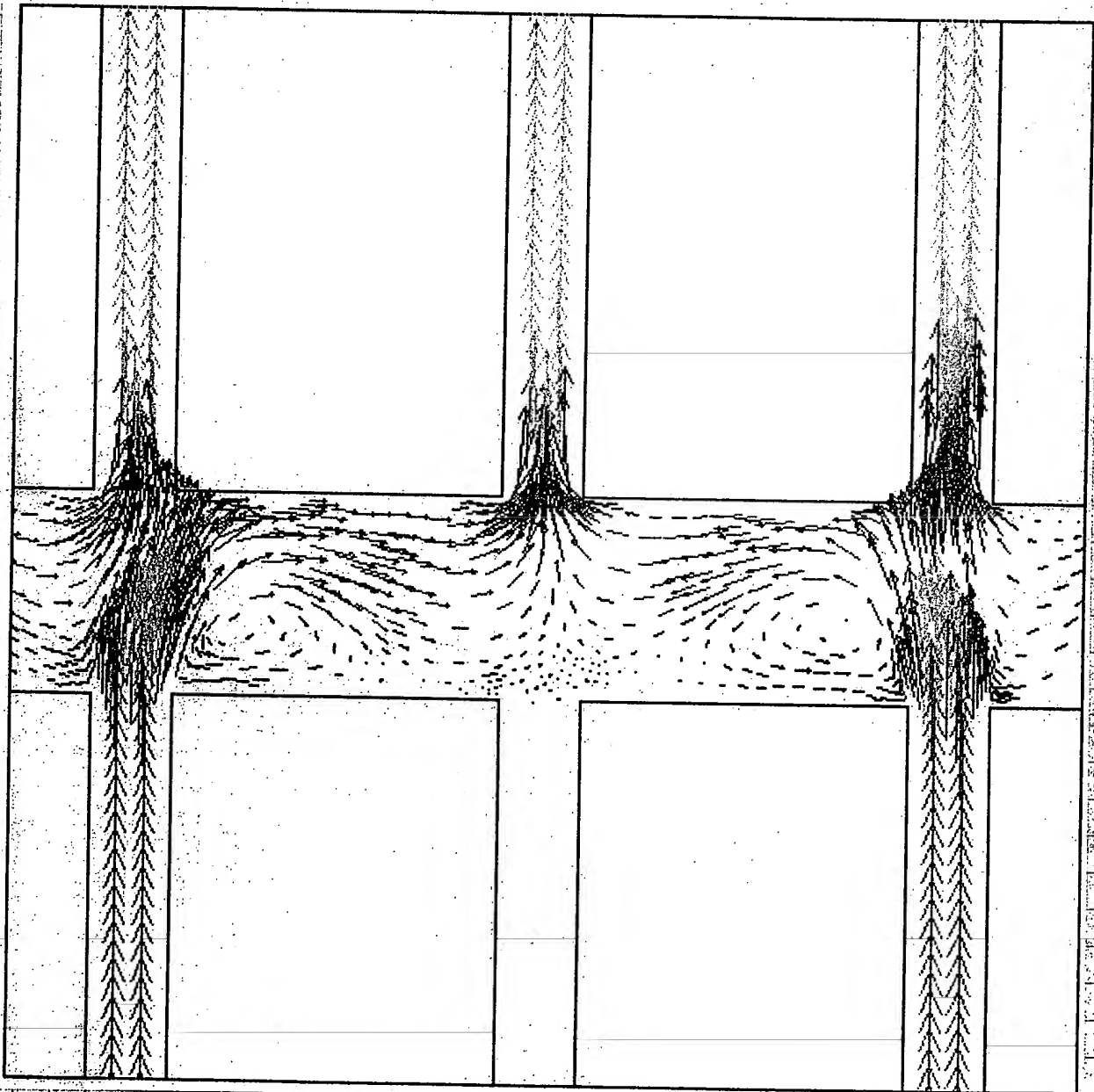
1.453

1.661



ANSYS 5.4
APR 8 1998
14:43:45
VECTOR
STEP=1
SUB =1
V
NODE=3675
MIN=0
MAX=1.459

0
.182355
.364711
.547066
.729421
.911776
1.094
1.276
1.459



in part of log

John D. [signature]

- assembly method attempted manually
under microscope using double-sided tape
150 μ m thick.

Results: Fair to poor!
25 needles arrays stacked
Still pursuing other methods.

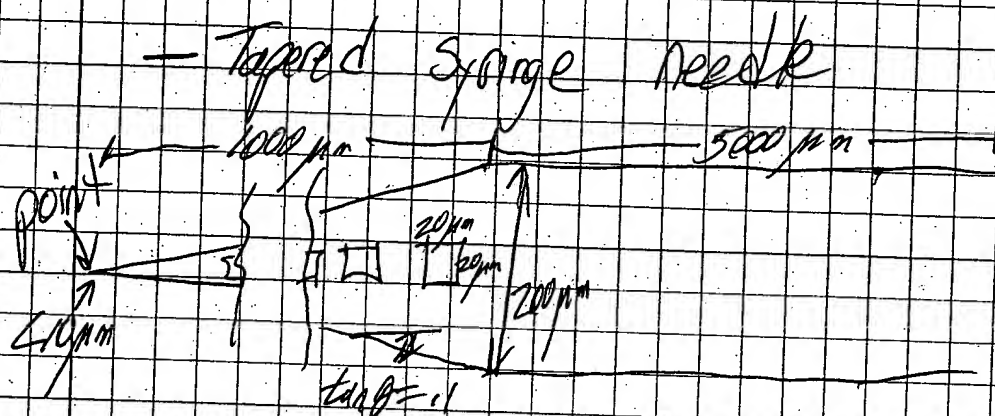
* Interface for single 2-D array is (746)
under manufacture.

List of items to be accomplished:

- 1- Bond 2-D array to single interface and
test w/ syringe.
- 2- Fabricate grid/mesh for assembly of
2-D arrays into a 3-D device.
- 3- Bond 3-D device to interface and
test w/ syringe

New Needle Design

(previous is considered old design)



Esage 919-990-2228

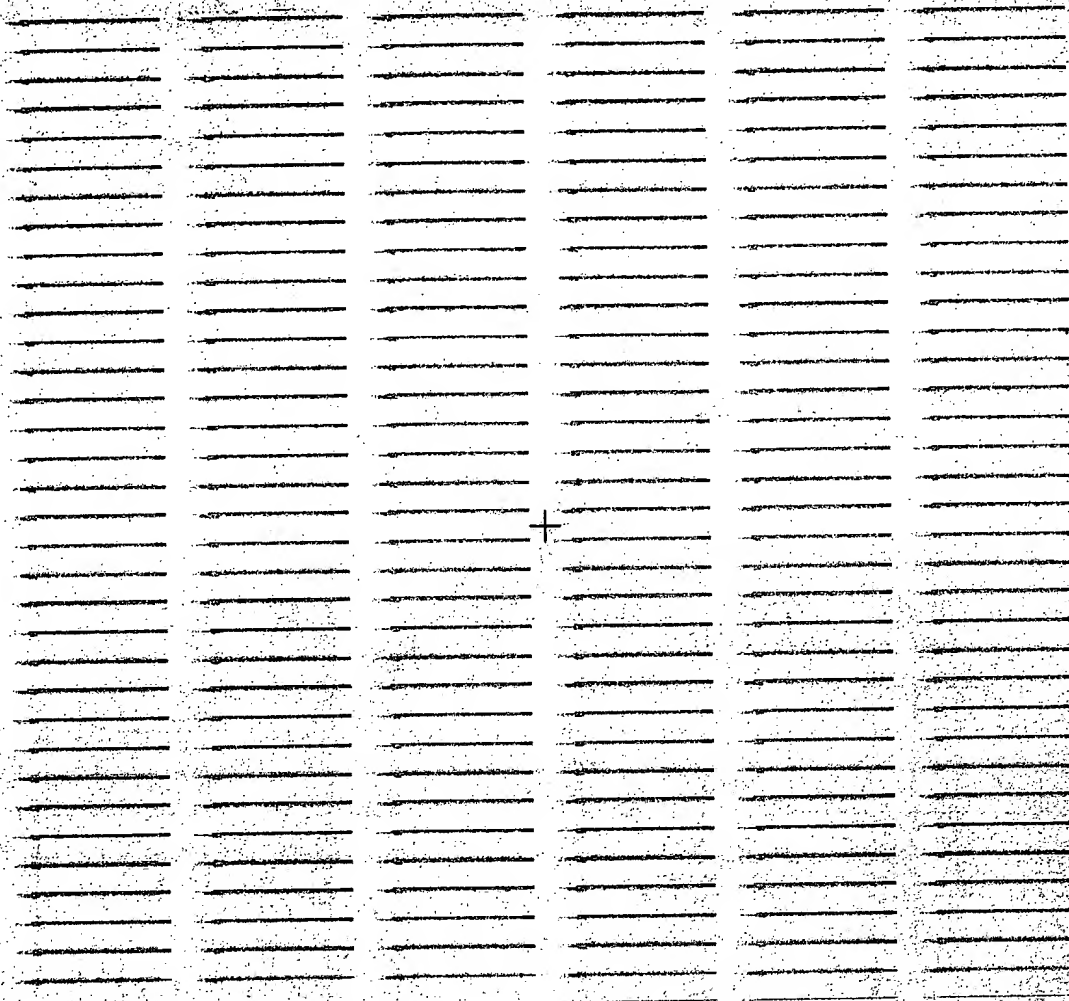
Notes:

- 1) Square cross section
- 2) along tapered section: place $20 \times 20 \mu m^2$ opening
at $d = 250, 300, 350, \dots, 1000 \mu m$
on two to four sides

9 July 98

Completed Mask Design

P. D. K.

↑
alignment
marks

Each needle is 6mm in length, 200µm in width.

Masks are completed,

6 columns of 35 each needles

Bottom shell area = ~~0.05~~ 0.55cm X 0.02cm X 35 X 6

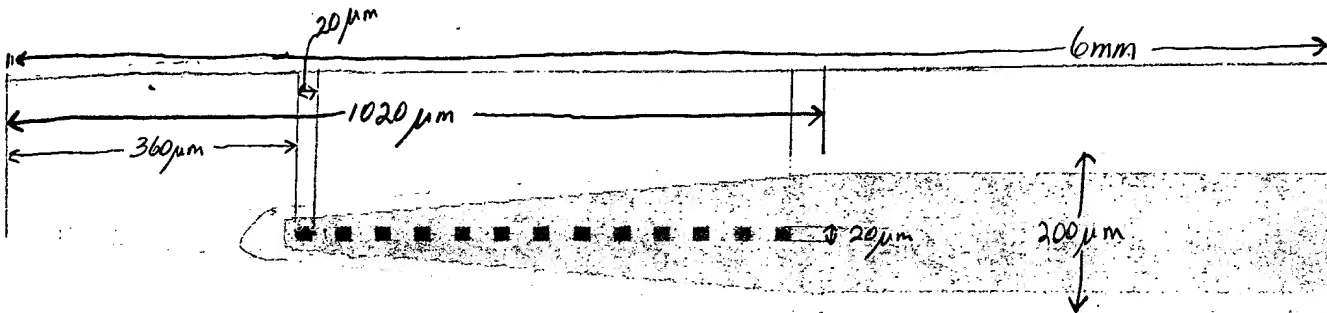
= 2.31 cm²

+ alignment marks

9 July 98

45

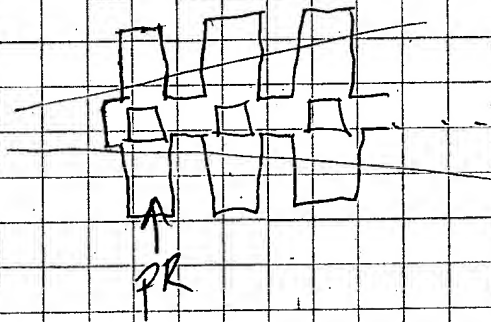
[Signature]
[Signature]



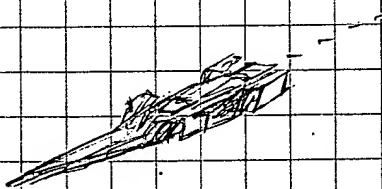
Note: approximately $20\mu\text{m}$ is expected to be lost on the needle tip length due to 'intentional' processing techniques.

This design allows for openings on two of the four sides.

For openings on four sides. Simply modify PR mask to:



should get:



9 July 98

Fabrication for lift-off of
new designs

Water prep

3" water

Clean:

4 min piranha-etch 70ml H_2SO_4 / 30ml H_2O_2
2 min DI rinse

30 sec HF (5%)
2 min DI rinse

4 min Metal-etch 33ml HCL / 33ml H_2O_2 / 33ml H_2O
2 min DI rinse

30 sec HF (5%)

2 min DI rinse

SO_2 on both sides ^{to ensure hydrophobic character} of water

Bottom shell formation

Spattered seed layers

Power: 250W

Argon: 60%

Vacuum: 1.2×10^{-6}

Pressure: 4.77

Flow: 55.7

Metals: Ti \rightarrow 4 min \rightarrow 1000 Å
Cu \rightarrow 3 min \rightarrow 2250 Å
Cr \rightarrow 3 min \rightarrow 1000 Å

Pattern Bottom Shell

PR 4620

300 rpm

15 sec

1000 rpm

20 sec

Bake 110°C for 2:15

expose: 45 sec

Develop 40 ml 400K / 100 ml H_2O

Results: 2nd part
some

10 July 98

John D. [Signature]

47

plate Bottom shell

etch Cr: 75% HCL

$Ni: 15 \text{ mÅ/cm}^2 \times 2.5 \text{ cm}^2 \approx 37 \text{ mÅ}$

Time: 1 hr \approx 17-18 min

$Pd: 5 \text{ mÅ/cm}^2 \times 2.5 \text{ cm}^2 \approx 12.5 \text{ mÅ}$

Time: 10 min

Stop PR in Acetone w/ alcohol rinse

Thick PR ON Bottom shell

PR 4/620

300 rpm 15 sec

1000 rpm 20 sec

bake: 110°C 2515

expose: 50 sec

Develop: 40 w/ 400K/100 w/ H_2O

Sputter Au

100 W DC for 3 min

substrate on top of foil

for heat dissipation

13 July 98

John D. Egan

Top shell Microemulsion

PR 4620

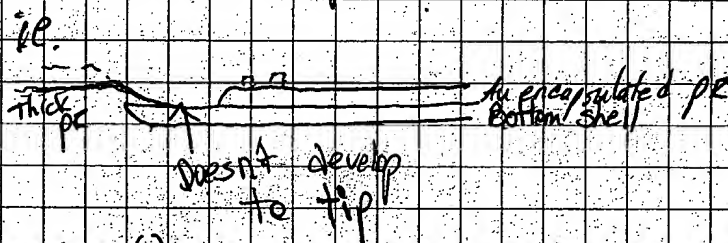
300 rpm
1000 rpm15 sec
20 sec

Cure in oven @ 37°C for 60 min

Expose 50 sec

Develop 40 ml 400X / 100 ml H₂O

Results: problem!

22 July 98
John D. Egan

Solution:

- Try Ashing @ 100 W O₂ RIE

- Change PR thickness to 10 μm

- spin @ 1500 rpm

- Maybe try thin PR just to see what happens

- offset mask for top-shell (compensation)

22 July 98

Phil [signature] 49

* prepared six more waters for trials (A-F)

~~Water~~ for piranha-etch 5 min
PT rinse 2 min
HF dip 30 sec
DI rinse 2 min

spin dry

deposit $Se_{1/4}$ both sides (40 min)

sputter Ti/Cu/Se 3/3/3 min

Trials:

Water A: 10 μm Bottom shell, 20 μm sacrificial PR, 10 μm Top shell

10 μm process: PR 4620 spin 1500 rpm 20 sec
cure & bake the same

Water B: ~~10 μm Bottom~~ same as water A but, ~~not change~~
~~change to 30 μm sacrificial PR (E-burn Air instead)~~
30 μm process (PR 4620 1500 rpm 20 sec) \approx 35 μm for top
shell and etch ~~after~~ after top shell/
micro mold is complete. This should force
plating to build-up on sides.

Water C: Thin PR, bottom shell, 20 μm sacrificial, Thin on Top
(plate 5 μm) (plate 5 μm)

Water D: 10 μm Bottom, 20 μm sacrificial, Thin PR Top
(plate 5 μm)
offset top-shell mask

* Waters E & F may be used for grid fabrication that will
be used in the assembly process of the old
needle design.

29 July 98

Wm V. Bzph

Top shell e-plating
on first two waters (Not trials yet)
plate around 5.1.1.1.2

- Strip
- Gold
- Aceto
- DI
- Resu



30 July 98

Wm V. Bzph

Trial water 'A & B'

10 pm Bottom shell

PL 4620 300 rpm 15 sec
1500 rpm 20 sec
bake 110°C for 2815

expose 40 sec
develop: 400K 40ml / H₂O 100 ml
Results: Very nice

MR

29 July 98

Top shell e-plating
on first two waters (Not trials yet)

plate around $5 \mu\text{m}$ @ $15 \text{ m}^2/\text{cm}^2$

- Strip PR

- Gold etch

- Acetone bath

- DI Rinse & dry

Results: - Both waters withstood high water flow without loss of adhesion.

- Microscope reveals fair appearance.

- Ready for SEM (later)

30 July 98

Trial water "A+B"

rpm Bottom shell

PL 4620 300 rpm 15 sec

1500 rpm 20 sec

bake 110°C for 2815

expose 40 sec

develop 400K 40ml H_2O 100 ml

Results: Very nice

31 July 98

John D. B. 51

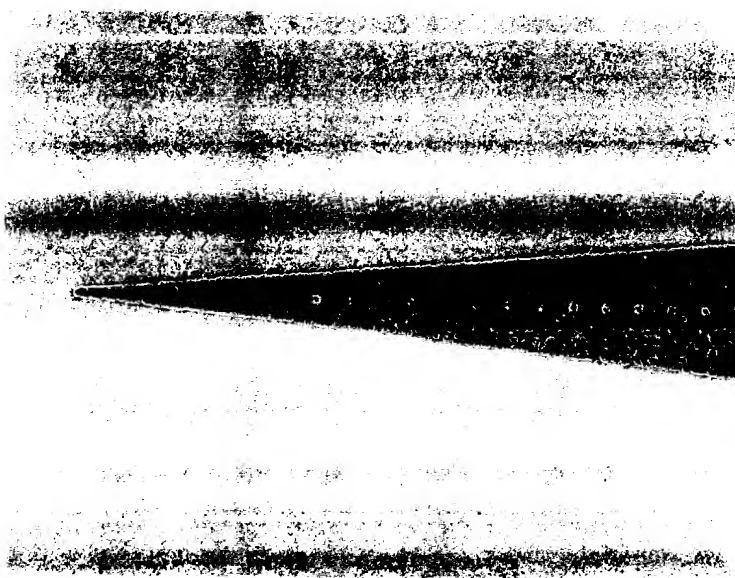
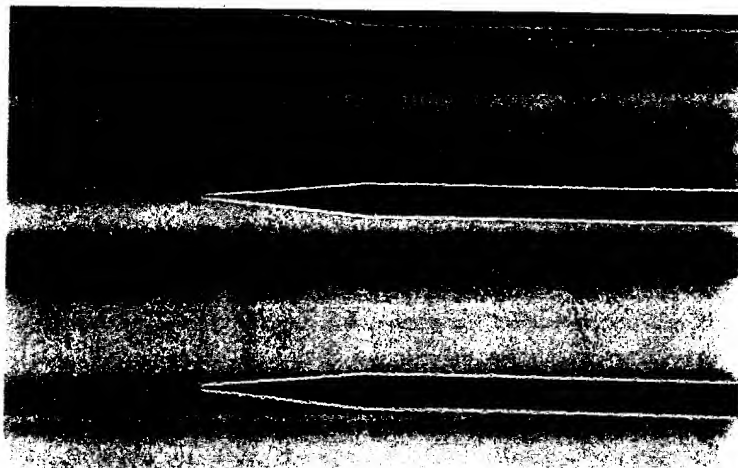
etch Cr in 25% HCl

plate Ni @ 36mA (5mA/cm²) for 35 min

plate Pd @ 36mA for 5 min

Results: very nice

strip PR in Acetone + methanol



Bottom
Shell $\approx 10\mu\text{m}$

3 Aug 98

John D.

Thick PR on bottom shell (Trials A+B)

need $\approx 20 \mu\text{m}$

PR 4620

300 rpm 15 sec
1000 rpm 20 sec

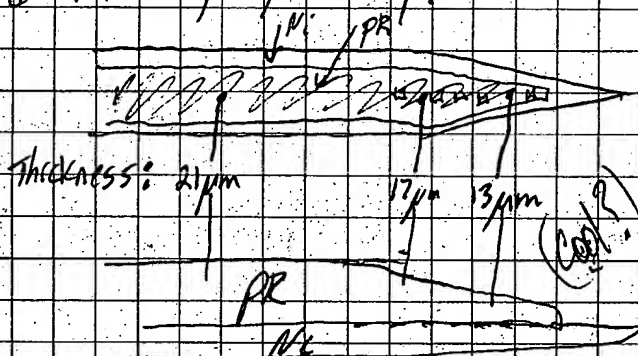
bake 110°C 245 min

expose 50 sec

develop 400K (40ml) / H₂O (100ml)

results: very nice

*note: on wafer A, development of PR down to chrome layer was not allowed. Development down to nickel bottomshell was targeted. Also, \approx measurement on hi-res microscope revealed following geometry:



3 Aug 98

~~John D. B.~~

00

Oil trial water A:

Sputter Au @ 100W for 3 min ~ 1000 Å
(water on heat-sink)

Results: Nice alignment, PR is wrinkled (try slow sputter next time)

" " water B:

E-gun Au for 1000 Å

Results: Alignment a little off, PR looks nice

Top shell micromold

10 Aug 98

~~John D. B.~~

Oil trial water A:

PR 4620

300 rpm
1500 rpm

15 sec
20 sec

~ 10 µm

bake @ 110°C for 1 hour

expose 50 sec

develop 40 ml 400K/100 ml H₂O

Results: GOOD!

Water B: PR 4620

300 rpm 15 sec

800 rpm 20 sec

~ 27 µm

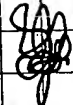
bake 110°C for 1.5 hours

expose: 65 sec

develop 40 ml 400K/100 ml H₂O

Results: GOOD!

mm



17 Aug 98

Philip

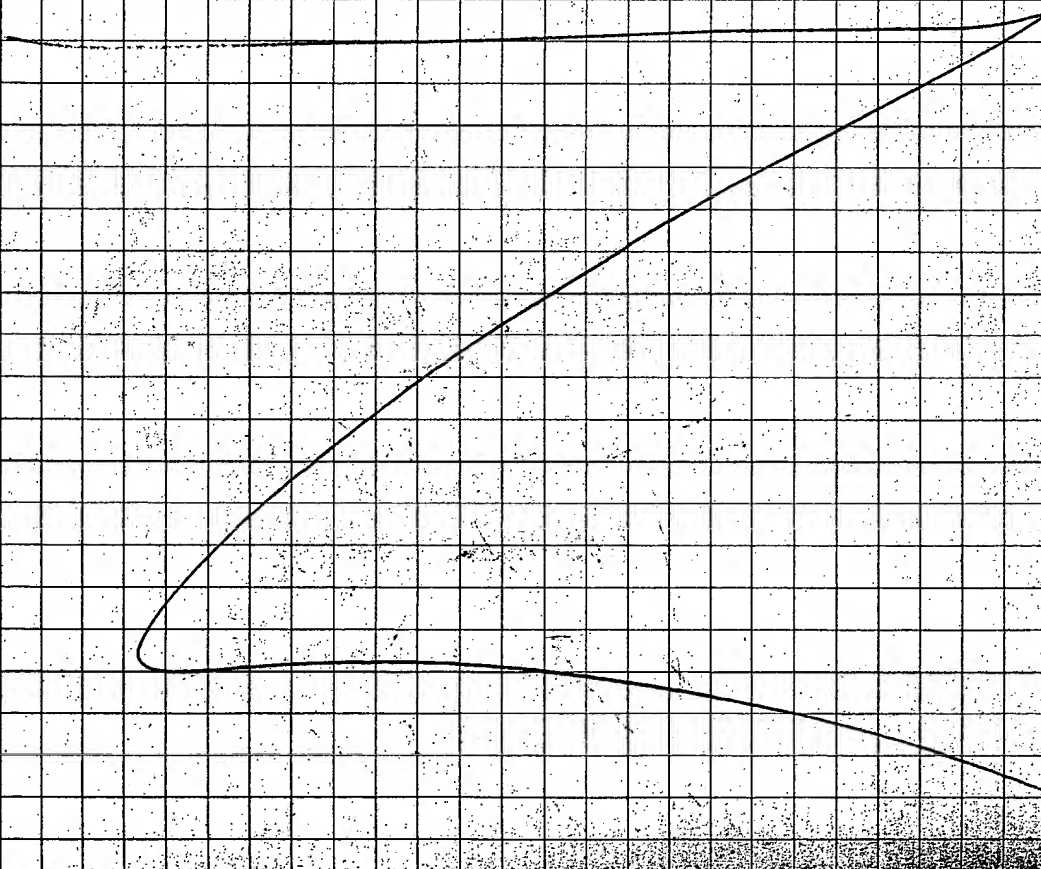
E-plate top shellwater B₂ (27 μ m micro-mold)16 bath @ 15 mA/cm²38 mA for 18 ~~hrs~~ 2 hours \approx 39 μ m

Results: parts covered-up

water A:

38 mA for 35 min

Results: Not good



10 Nov 98

New Batch!

[Signature]

55

12 wafers pgh and coated w/oxide + Ti/Cu/Cr

Per 3
Bottom shell $\approx 10 \mu m$

PR 4620 300 rpm 15 sec
1500 rpm 20 sec

bake 110°C for 2:15 min

expose 40 sec

develop 400K 40 ml / H₂O 100 ml

Results: GOOD!

plate: etch Cr in 75% HCl

Ni @ 38 mT (15 mT/cm²) for 35 min

Pd @ 12.5 mT (5 mT/cm²) for 10 min

strip PR + rise

Results: GOOD

12 Nov 98

Sacrificial layer

add 6 min Ash in O₂ (15 min)

PR 4620 300 rpm 15 sec
1000 rpm 20 sec $\approx 21.7 \mu m$
bake 110°C 2:15

expose 50 sec

develop 400K 40 ml / H₂O 100 ml

Results:

Sputter Au @ 50 W for 6 min - 1000 Å
(water on heat sink)

Results: WAVES in PR (encapsulated)

13 NOV 98

TOP SKULL

QR 4/620

300 rpm
1500 rpm15 sec
20 sec

Bake @ 400°C in oven for 1 hour

Exposure: 50 sec @ 55kV

Develop 40 ml D19 / 40 ml D19

Results: GOOD w/exception of alignment

E plate:

Two waters in Ni @ 38 mA (5 min) to 3 min

Strip PR (under developed)

Results: ~~GOOD~~ ~~Adhesion~~

Oil water in Pd @ 12.5 mA (5 min) for 2 min

Strip PR

Results: GOOD Adhesion

~~LIST OF~~~~Good each x 30 sec~~~~5 min~~~~2 min~~~~Crack in~~~~Cu plate~~~~Results:~~~~SEM TIME:~~

13 Nov 98

Start new Batch using preceding
Changes! Do on Monday

57

17 Nov 98

Also add placement of water
into ultrasonic water bath prior
to top-shell e-plate.

Another Batch

Use process on pgs 55-56
unless otherwise noted.

Bottom shell $\approx 10 \mu\text{m}$ GOOD!

23 Nov 98 Sacrificial layer w/ 5 min 30% O_2 added

Sputter changed to 1 min ON and 5 min OFF
and cycled three times.

Results: Alignment Problems!

24 Nov 98 Top shell add 5 sec & overlap longer

Results: looks pretty good
still have alignment problem

e-plate Results: GOOD, but missing

Note: tip of hollow portion of needle
stripping top shell OR to redo for
21.2 μm thick specs.

17 Dec 98

Results on

Thick Top shell PR (2)

Process:

PR 4620 300 rpm 15 sec

1000 rpm 20 sec

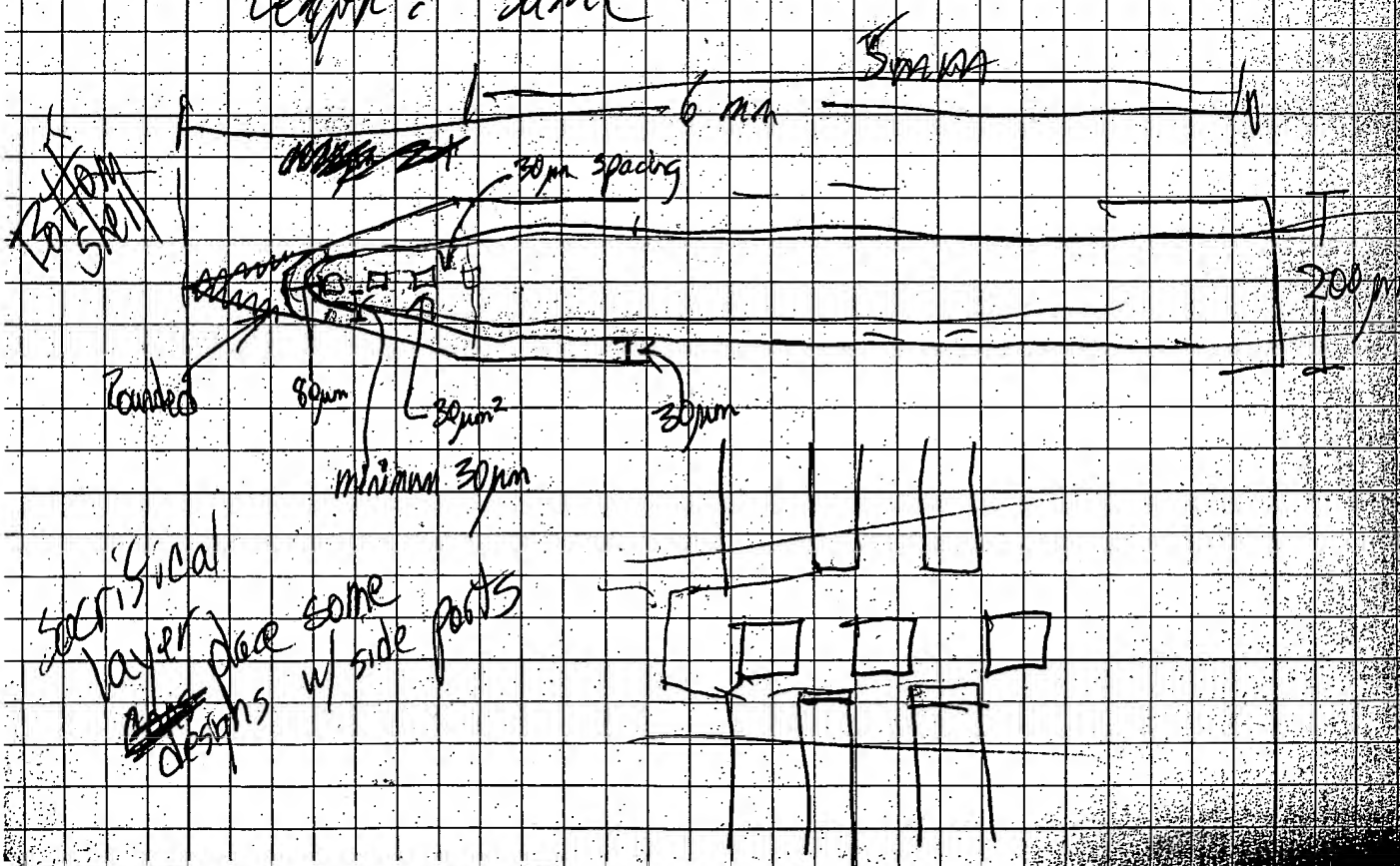
Bake in oven 50°C 1 hour

Exposure 00 sec

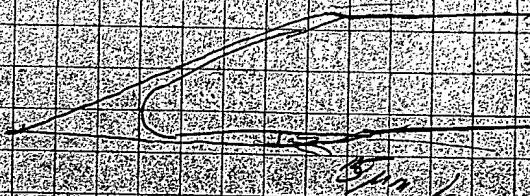
18 Dec 98

Redesign Mask!

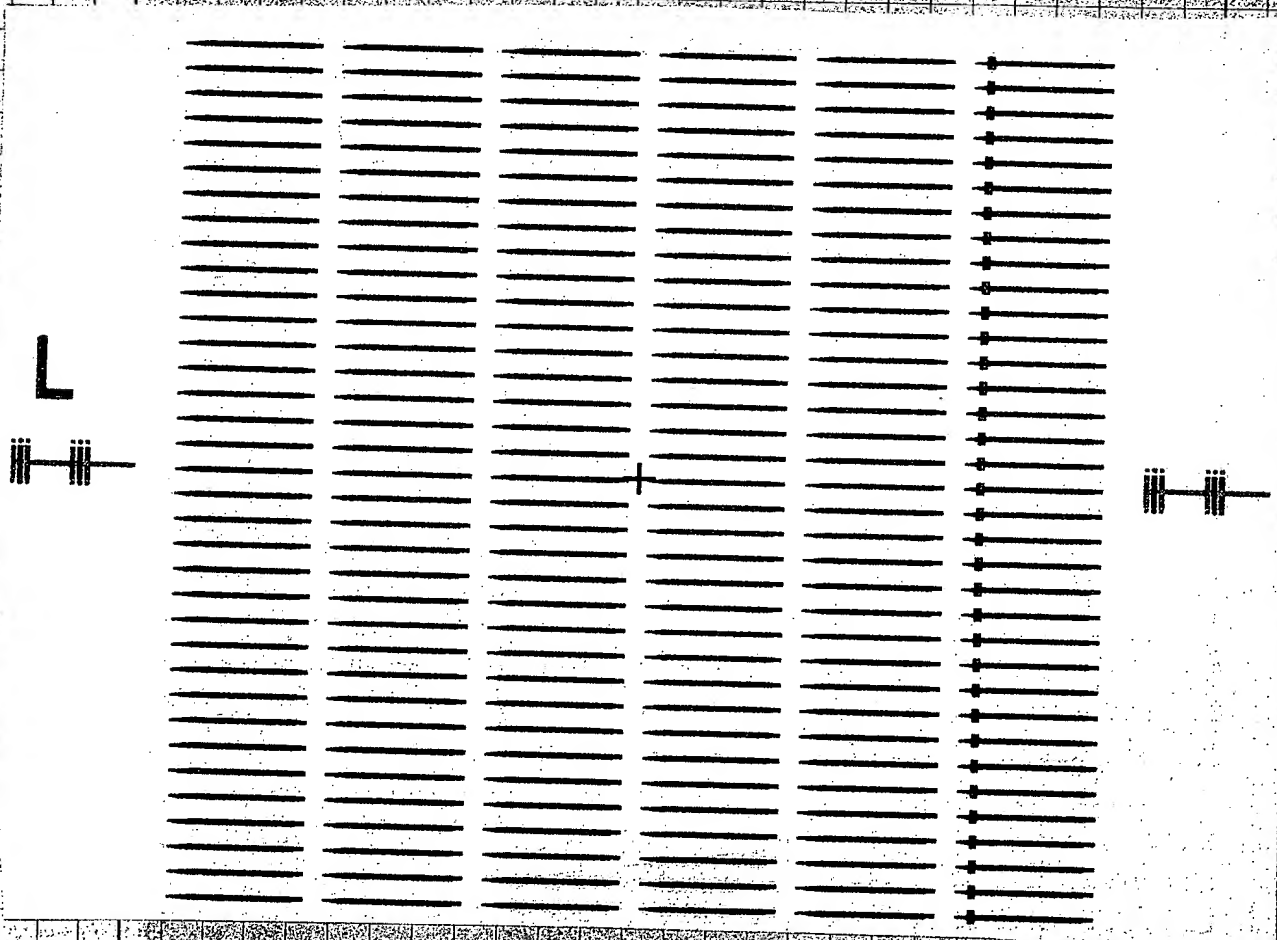
Length: Same



Top shell



6 Jan 98 ~~11~~ Completed Nest Design



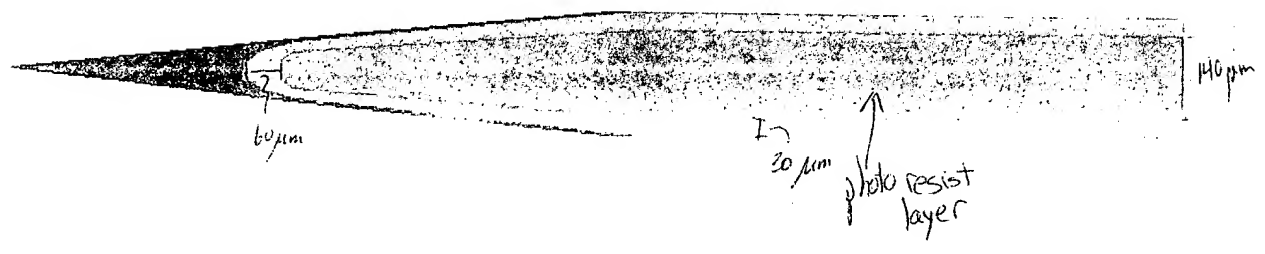
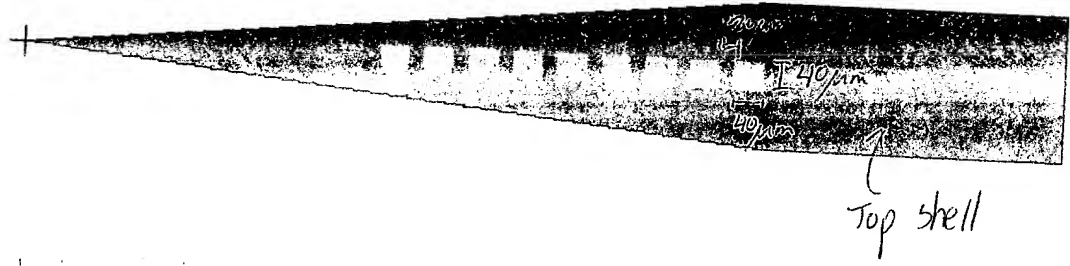
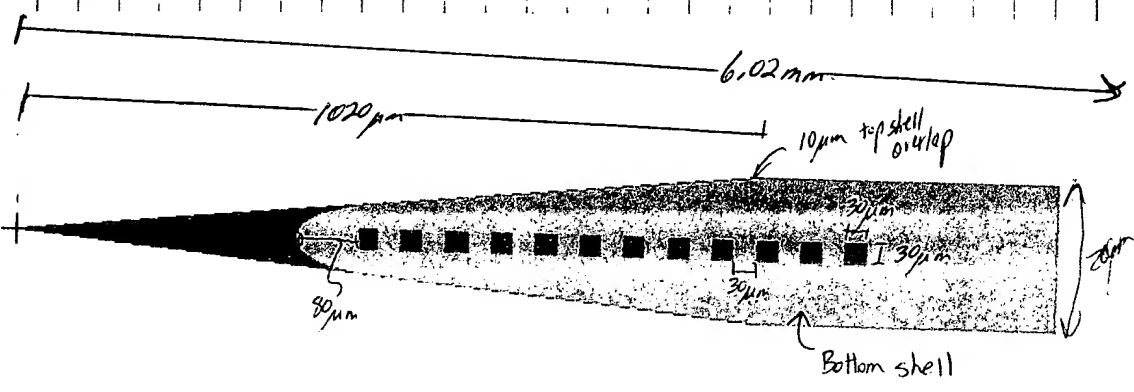
Layer 46
Layer 49
Layer 43

Platensis
Top shell
Bottom shell

This column
contains side-point design

11 Jan 98

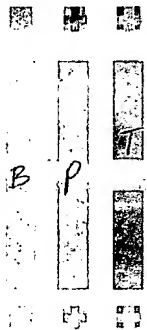
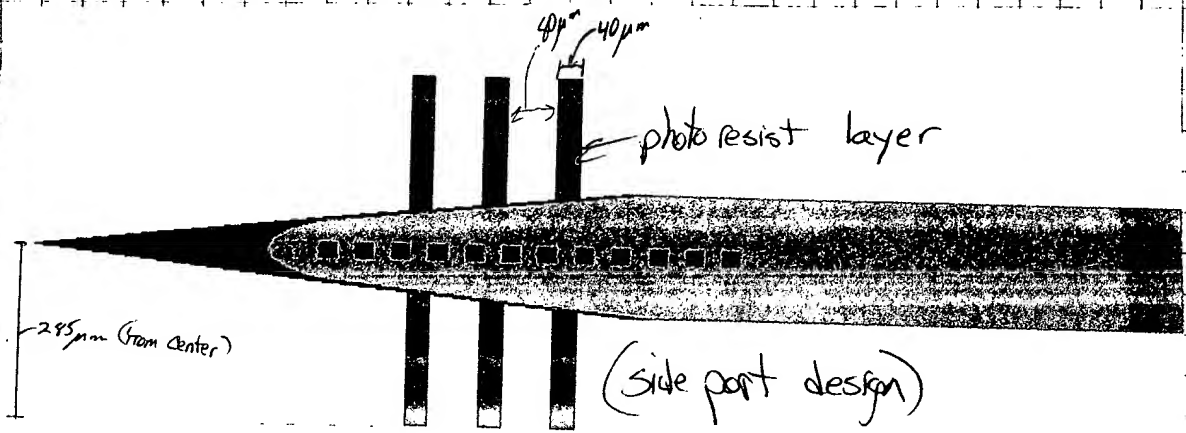
[Signature]



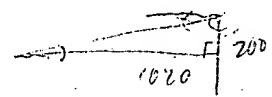
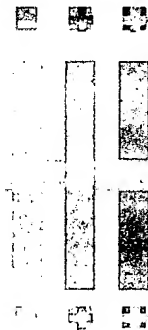
11 Jan 98

John D. [Signature]

61



Alignment marks
(left)



20 Jan 99

All checks in hand!

Clean waters w/ Ti/Cu/Er

Bottom shell $\approx 10 \mu\text{m}$

PR 4620

300 rpm

15 sec

1500 rpm

20 sec

 $\approx 12 \mu\text{m}$

Duke NORC hot plate for 2.5

expose 40 sec

develop: 400K 40% H_2O 100 ml

Results: GOOD!

e-pate

Ni @ 37 mA for 35 min $\approx 10 \mu\text{m}$ Pd @ 12.5 mA for 10 min $\approx 1 \mu\text{m}$

Strip PR w/ acetone + methanol

Results: Peel-off!

Either poor Cu layer or need
to stay longer in HCL!

21 Jan 99

63

Sacrificial Layer

PR 4620

300 rpm 15 sec

1000 rpm 20 sec \approx 21 μ m

bake 110°C for 2:15

expose 50 sec

develop 400K

Results: Not Bad

~~watch~~ watch for overdeveloping

E-gun Au \approx 1000 Å

* problem w/ pressure during evaporation.

Top shell

PR 4620

300 rpm 15 sec

1000 rpm 20 sec

Bake in oven @ 50°C 1 hour

expose: 60 sec + develop 400K

Results: GOOD

Note: PR of sacrificial layer
is being etched.

E-plate Ni @ 37mA for 1 hour

* Plating on top of sac layer occurred immediately.

Results: looks pretty good, need SEM
to verify side-walls!

64
29 Jan 99

started another Rev

- pattern bottom shell

- e-plate \approx comm

Results: very nice! use $\frac{1}{4}$ second
ultra sonic bath

4 Feb 99

- Sacrificial layer followed by 5 min ash

Results: GOOD appearance from top

- Sputter Au: 50V for 1 min
 $\frac{1}{4}$ 5 min cool
(3 cycles)

Results: very nice!
NO waves.

5 Feb 99

top shell

PR and bake

expose 60 sec & develop

Results: GOOD.

6 Feb 99

e-plate

110V @ 37 mA for 1 hour

Results: GOOD.

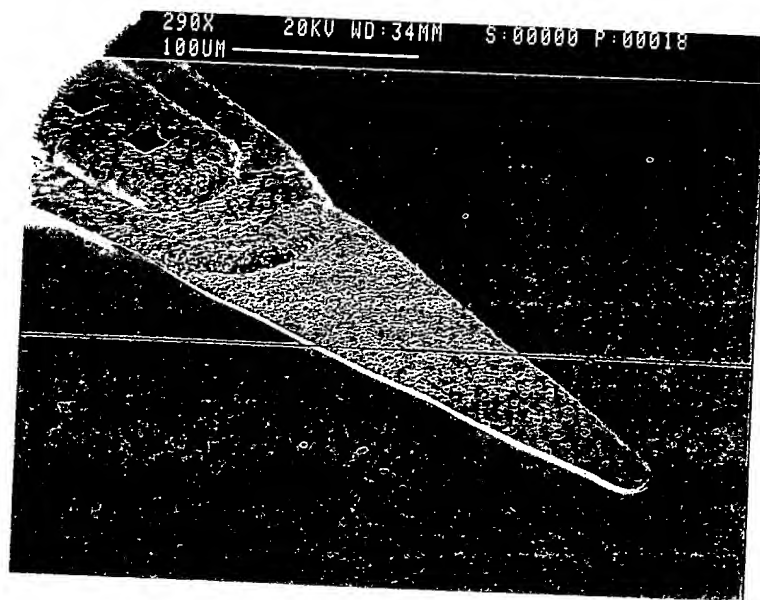
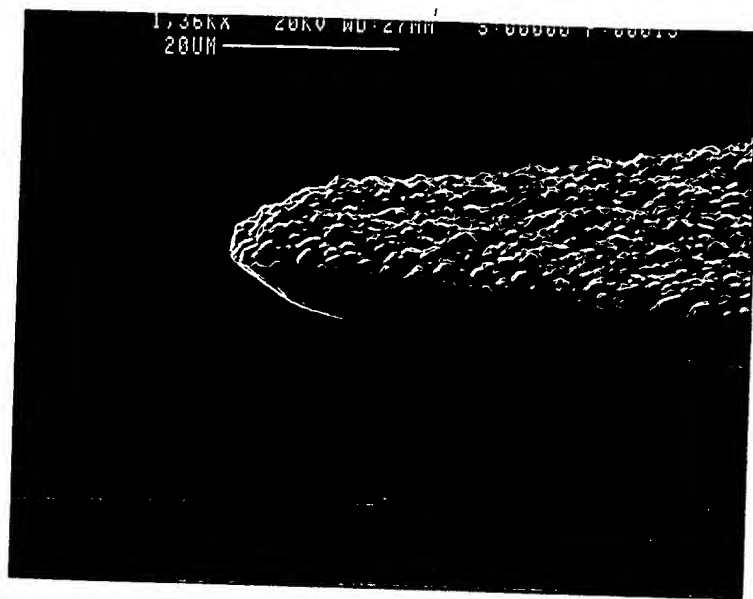
17 Feb 77

Page 1 of 1

03

SEM: Looks pretty good

7/31



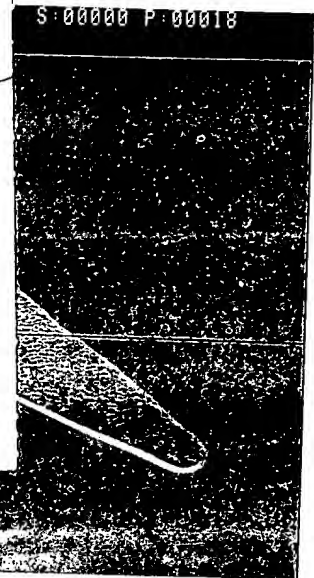
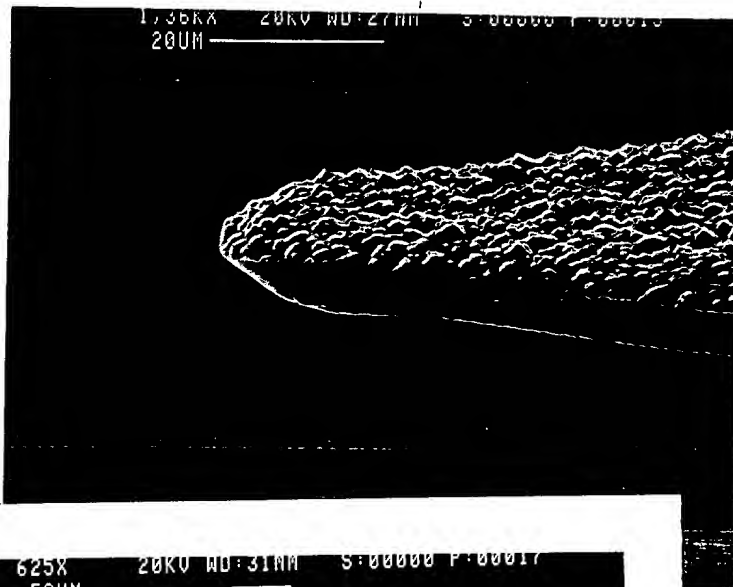
Polaroid H62724170418C

14 Feb 94

65

SEM 8 Looks pretty good

7/31



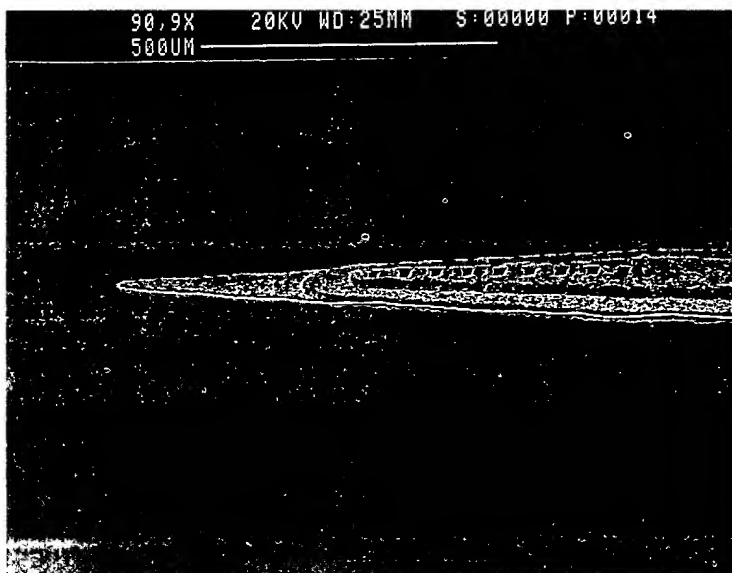
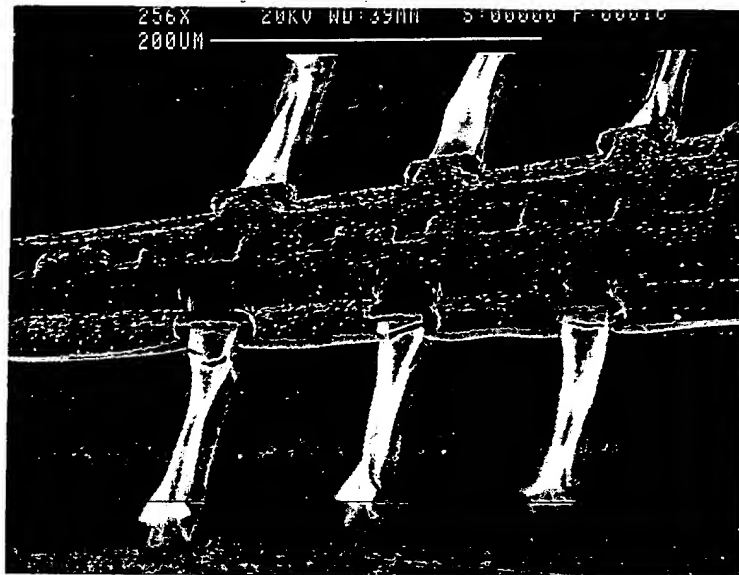
66

200X

11 Feb 99

Mr. O. King

SEM



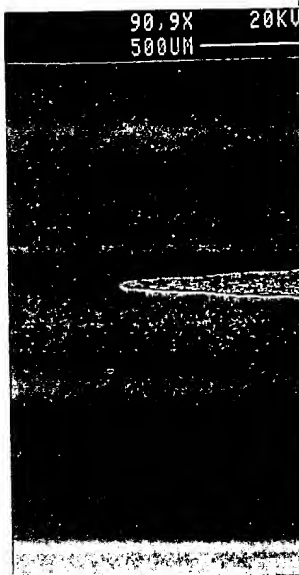
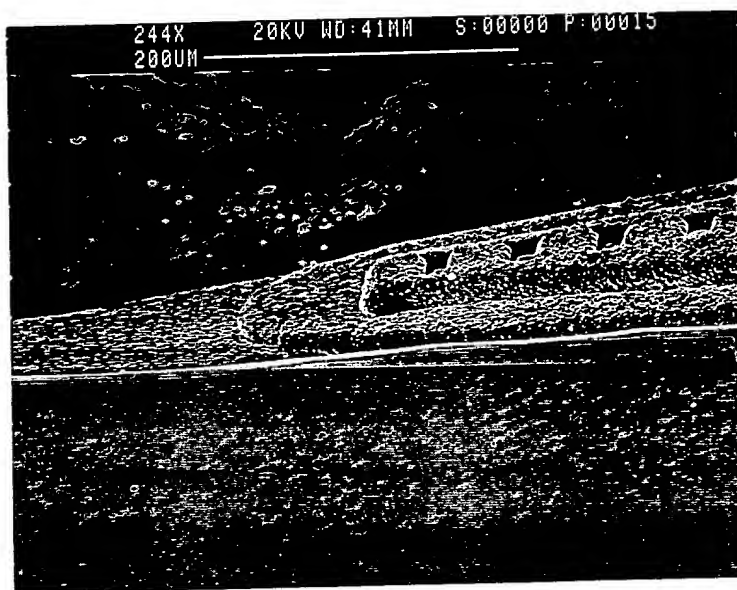
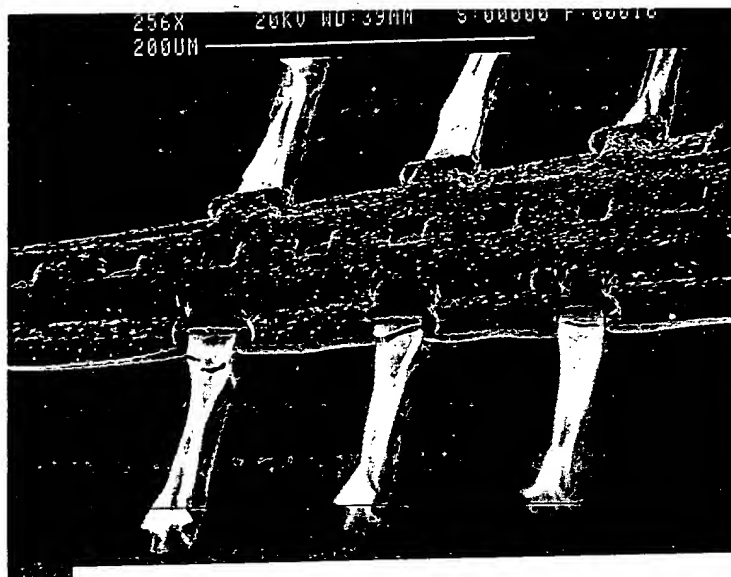
66

AWK

11 Feb 99

Handwritten signature

SEM



11 Feb 99

67

Release Needles

- 1- 1 sec in ultra pure bath of DI water
- 2- 15 sec Au-etch
- 3- 10 min Acetone
- 4- 5 min 2-propanol

5- Cr-etch 1 min

6- Ammonium Hydroxide saturated w/ cupric sulphate
1.5 hours

Results: ~~Good~~

* Ended up using the yellow Cr-etch. Surprisingly it removed the Cu layer in under a minute without damaging the nickel.

68

17 Feb 99

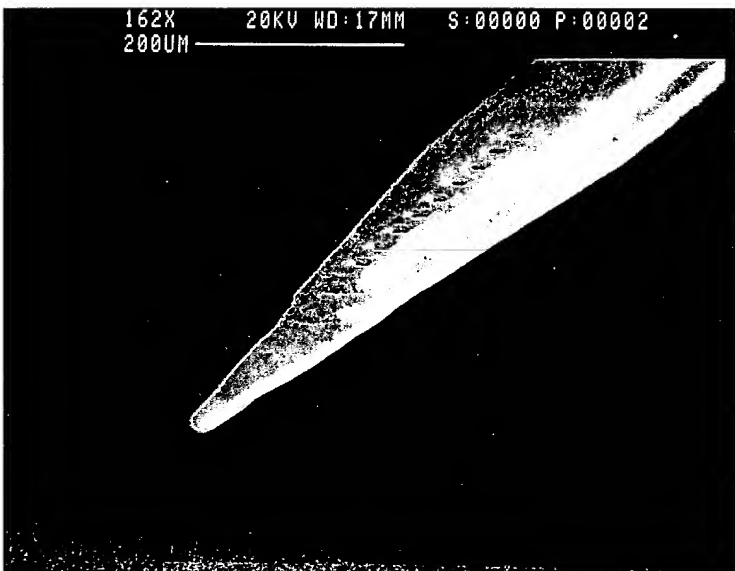
John D. Vigg

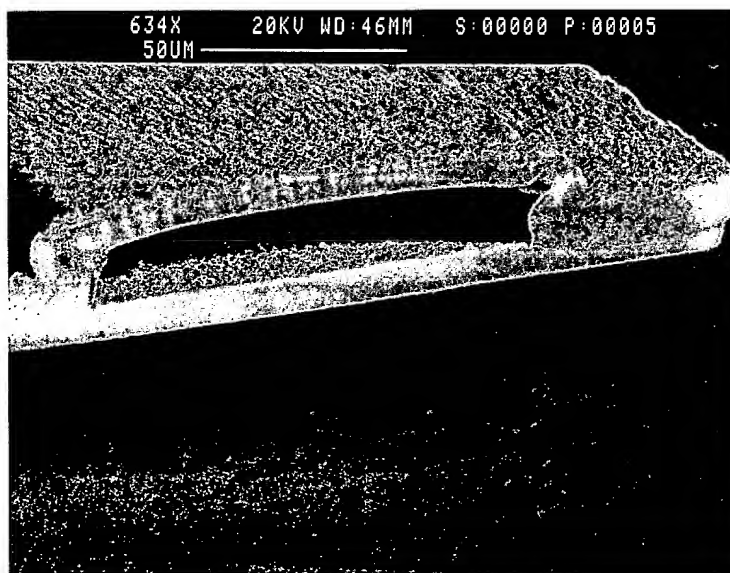
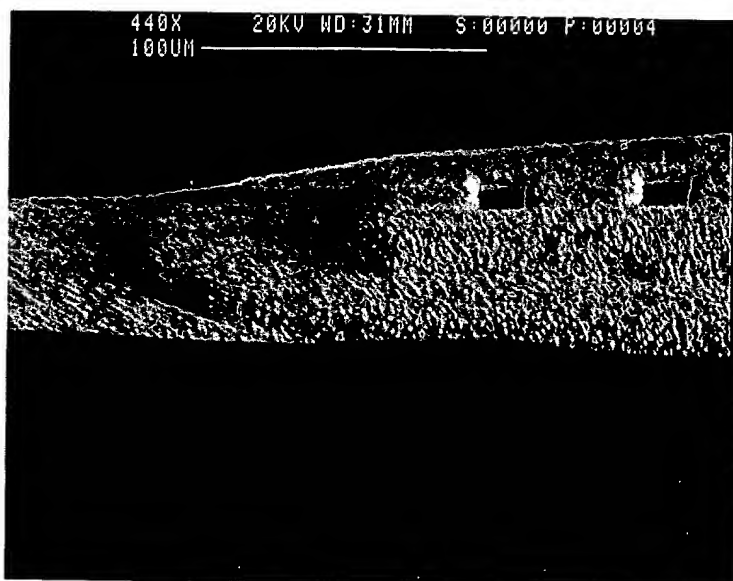
Released the nicer of the
two waters & mounted for
SEM.

23 Feb 99

Results GOOD!

John D. Vigg





1 Mar 99

Attempts to thicken ~~bottom~~
shell!

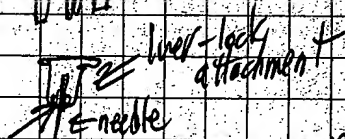
Can go 20mm as thick as I
without losing parts!

3 Mar 99

packaging



← syringe



← needle

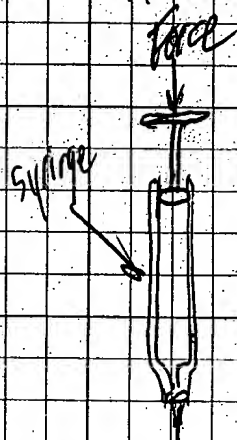
will
cure
adhesive

luer-lock
attachment

need pictures!

11 Mar 99

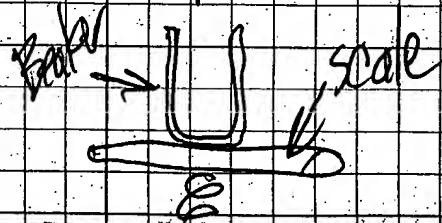
Testing



syringe

force

* apply constant force over
period of time and
measure mass of
water.



scale

11 Mar 99

71

Preliminary testing results

Max: -9.95 } cycle speed: 10 mm/min
Min: -10 }

Run 30 min \Rightarrow Mass₄₀: 1.3932g

* Experiment w/ speed to get best
constant force.

16 Mar 99

Needle Testing

Max: -9.975 } cycled speed: 10 mm/min
Min: -10.000 }

① ~~30~~ 30 minute runs: needle ①

1 - .7194g

2 - .6713g

3 - .6172

4 - .9458

5 - .7606

6 - .7487

7 - .7615

15 Mar 99

Testing from yesterday

8-0.7578

9-0.7364

10-0.7563

at 20 lbs force needle @
max: 19.97 min: 20

✓ 2.3304 g

✓ 2.6946

✓ 2.8363 ch 15 min runs

15 Mar 99
Needle @

30 Mar 99

Force → pressure conversion

 $d = .485$ by micrometerArea of syringe plunger: $\frac{\pi d^2}{4} = \frac{\pi (.485)^2}{4} = .1847 \text{ in}^2$

pressure scale

Force 100 psi → F = 18.47	16 → 7.388	1 → .1847
90 → 16.623	30 → 5.541	.5 → .09235
80 → 14.776	20 → 3.694	.1 → .01847
70 → 12.929	10 → 1.847	
60 → 11.082	5 → .9235	
50 → 9.235	4 → .7388	
	3 → .5541	
	2 → .3694	

30 Mar 99

[Signature]

~~10 psi~~

min: -1.005
max: -.9995
speed: 10

for 30 minutes

- 1- .0035
- 2- .0066
- 3- .0053
- 4- .0065

1 lb F \Rightarrow > 5 psi

Needle III

1 psi

min: -.850
max: -.845
speed: 5

for 30 minutes

Needle IV

- 1- .0052
- 2- .0058

gravity!

for 30 minutes

- 1- .0048
- 2- .0046

10 psi

min: -1.850
max: -1.845

speed: 10 30 min

31 Mar 99

[Signature]

1- .0064

2- .0073

20 psi

min: -3.700
max: -3.690

speed: 10 30 min

1- .0175

2- .0606

3- .0404

4- .0381

5- .0279

6- .0194

6

1 Apr 99

30 psi

MIN: -5.545
MAX: -5.535

speed 2/10

30 min

1- .1290

2- .1156

3- .1230

4- .1217

Flow calculation

 $m = dV$ $d_{H_2O} = 1 \text{ g/cm}^3 @ \text{Room Temp}$

$$\therefore V = \frac{.1230 \text{ g}}{1 \text{ g/cm}^3} = .1230 \text{ cm}^3$$

$$\text{Rate} = .0041 \text{ cc/min} = .246 \text{ cc/sec}$$

13 Apr 99

All data previous is wrong

30 psi

MIN: -5.545

MAX: -5.535

speed

15

30 min

1- 4.1514 g

evap rate: 4.1501 over 3 min
4.1485

2-

sprung leak

1 psi

min: -1.850

max: -1.843

speed

2

30 min

1- .0035

5- .0060

2- .0033

6- .0050

3- .0074

7- .0055

4- .0056

8- .0067

Need it

$$\Delta W = \pm .0001$$

$$\Delta F = \pm .5\%$$

$$\Delta d = \pm .0005"$$

$$\Delta t = .015$$

$$= .001667 \text{ min}$$

$$W =$$

$$F =$$

$$d = .485$$

$$t = 30 \text{ min}$$

$$m_{\text{final}} =$$

$$m_{\text{avg}} =$$

$$\Delta m_{\text{final}} \approx .0001$$

$$\Delta m_{\text{avg}} \approx .0001$$

$$\Delta P = \left| \frac{\partial P}{\partial F} \right| \Delta F + \left| \frac{\partial P}{\partial d} \right| \Delta d$$

↑ sensitivity coefficients

$$= \left| \frac{1}{\pi d^3/4} \right| \Delta F + \left| \frac{2F}{\pi/4 d^3} \right| \Delta d$$

$$= \left| \frac{1}{\pi d^3/4} \right| \Delta F + \frac{2F}{\pi d^3/4} \Delta d$$

$$\Delta P = \frac{1}{\pi (.485)^3/4} (F)(.0005) + \frac{2F}{\pi (.485)^3/4} (.0005)$$

.151 high

.014 low

$$\Delta P = .0825$$

$$P = 20 \pm .05 \text{ psi}$$

$$Q = \frac{m_{\text{final}} + m_{\text{avg}}}{t}$$

$$\Delta Q = \left| \frac{\partial Q}{\partial m_{\text{final}}} \right| \Delta m_{\text{final}} + \left| \frac{\partial Q}{\partial m_{\text{avg}}} \right| \Delta m_{\text{avg}} + \left| \frac{\partial Q}{\partial t} \right| \Delta t$$

based on instrument reading
actual uncertainty may be higher

$$\frac{m_{\text{final}} + m_{\text{avg}}}{t} \times .001667$$

High 1.55×10^{-5}

Low 6.68×10^{-6}

$$\Delta Q = .000011$$

12 May 99

[Signature]

75

1psi

Min: -.1850

Max: -.1845

speed: .2 for 30 min

Runs: 1. .0215 (slow) 6. .0051
2. .0044 7. .0056
3. .0049 8. .0058
4. .0051 9. .0064
5. .0049 10. .0052

2psi

Min: ~~.3700~~ .3700

Max: -.3690

speed .2 for 30 min

Runs: 1. .0044
2. .0060

13 May 99

[Signature]

3. .0059

4. .0054

5. .0050

3psi

Min: -.5545

speed .2 @ 30 min

Max: -.5535

1. .0050 (same as previous)

5psi

Min: -.9240

Max: -.9230

17 May 94

Needle VI

3 psi Min: -.9240 @ .2 for 30 min
 MAX: -.9230

1-.0134 6-.0089

2-.0086 7-.0100

3-.0111 8-.0097

4-.0098 9-.0090

5-.0091

18 May 94

10 psi Min: -1.855 @ .2 for 30 min
 MAX: -1.840

1-.1332 6-.0761

2-.1140 7-.0707

3-.0980 8-.0673

4-.0739 9-.0656

5-.0727 10-.0416

20 June 99

10 psi

min: -1.853

max: -1.840

0.2 for 30 min

1- .0483

6- .0343

2- .0687

3- .0644

4- .0326

5- .0321

18 June 99

10 psi again

1- .1454

21 June 99

2- .1137 5-

3- .0481

4- .0292

20 psi

min: -3.07

max: -3.69

0.2 for 30 min

1- .07738

~~23~~ 23 June 99

2- 1.2024

3- 1.1225

4- 1.1587

5- 1.0999

6- .9635

7- 1.1787

29 Jun 99

30 psi - 5.545 = min 0.2 for 30 min
 - 5.535 = max

1- 1.6928 3- 1.7028

1 July 99 4- 1.6135

2- 1.6215 5- 1.6842

40 psi - 7.390 = min 0.2 for 30 min
 - 7.380 = max

1- 2.2054 3- 2.1089 5- 2.4018

2- 2.3402 4- 2.2531 6-

50 psi - 9.230 = max 0.2 for 30 min
 - 9.240 = min

1- 3.0630 3- 3.2218 5- 3.1554

2- 3.1811 4- 3.0528 6-

60 psi - 11.08 = max 0.2 for 30 min
 - 11.09 = min

1- 4.1964 3- 4.2154 5- 4.1332

2- 3.9776 4- 4.0345 6-

70 psi - 12.935 = min 0.2 for 15 min
 - 12.985 = max

1- 2.4535

2- 2.3365

19 July 99

79

Goal: Make needles thicker!

Clean wafers w/ Ti/Cu/Cr

Bottom shell $\approx 17\mu\text{m}$ of Ni

- PR 4620

300 rpm

15 sec

1000 rpm

20 sec

bake 110°C for 2:15 (yields $24\mu\text{m}$ mold)

exposes 10 sec

develop: 400K (40ml) / H_2O (100ml)

Results: maybe slightly overexposed?

(.5 sec ultrasonic bath prior to cr etch)

- etch Cr in 25% HCL

~~1 Aug 99~~

Ni @ 37mA for 1 hour $\approx 18\mu\text{m}$

Pd @ 125mA for 10 min $\approx 1\mu\text{m}$

strip PR w/ acetone + nitroal

Results: GOOD but output ports are covered

~~25 Jul 99~~

Sacrificial Layer

PR 4620

300 rpm

15 s

1000 rpm

20 s

bake: 110°C for 2:15

exposes: 10 sec

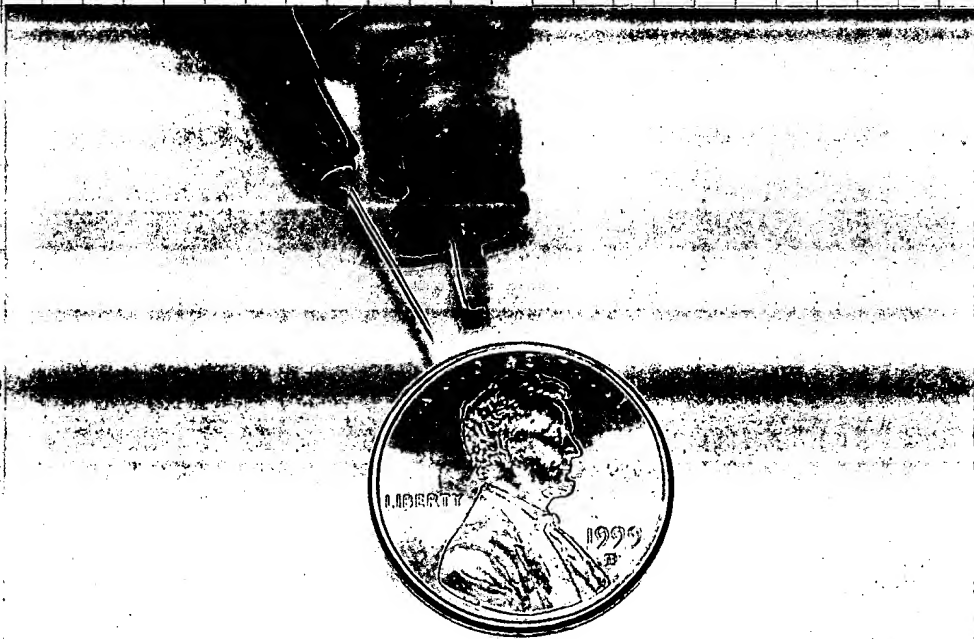
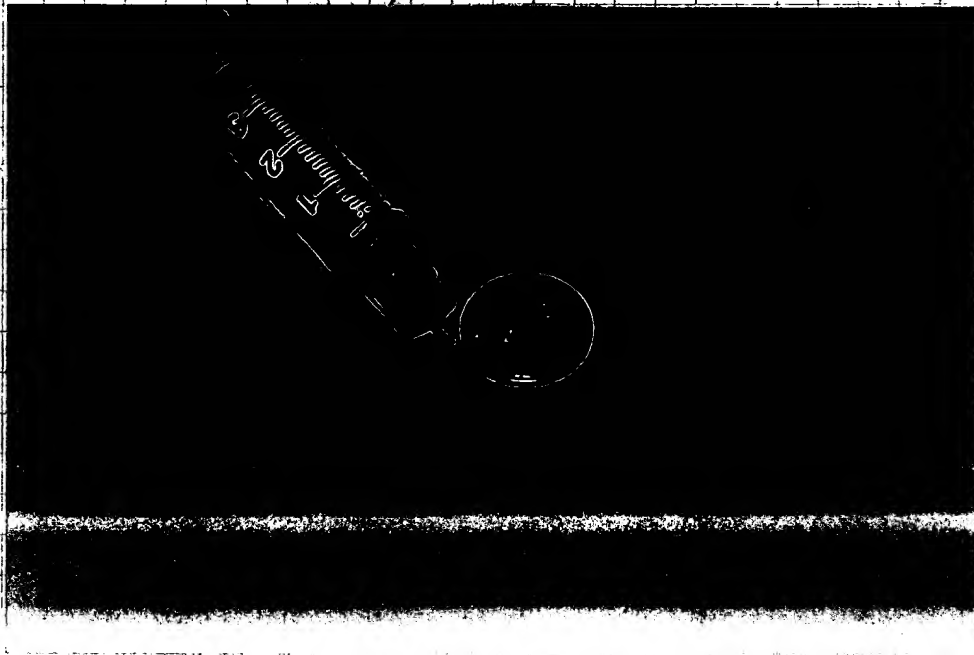
develop: 400K 40ml / H_2O 100ml

Results:

Pictures of packaged needle!


15 x 94

W. & W.




1541 94 *John D. Brazzle*

... travel grant, and (b) proposed budget.



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